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Does it Matter?

WHEN an engineer works out his designs for a structure or a component of a machine, he makes use of a "factor of safety" or, as it may equally appropiately be called, a "factor of ignorance," which makes allowance for the uncertainties which are inherent in his calculations. These uncertainties may arise in several ways. They may spring from the difficulty of assessing the range of conditions likely to be met in service, or from the snags encountered in determining exactly what stresses will be applied as a result of a particular set of operating conditions. On the other hand, they may have their origin in the "unreliability" of the materials of construction. As was pointed out by Mr. Frank Hudson in introducing his paper on "The Influence of Specifications on Productivity and the Economic Utilisation of Ferrous and Non-ferrous Metals," at the Institute of Metals Discussion on Metal Economics, accurate correlation between test-bar results and the mechanical properties of the casting itself, together with control and uniformity of quality, would enable many castings to be redesigned, with a resultant saving of metal, due to the lower factors of safety necessary.

We take this opportunity of extending to Readers our Cordial Greetings for the Festive Season and Good Wishes for the Coming Year.

In recent issues, we have had occasion to refer to the various ways in which efforts are being made to ease the metal shortages prevailing at the present time. The more obvious steps, such as prohibiting the use of strategic materials for luxury purposes, and encouraging the collection of scrap and its use in secondary metal, etc., have already been taken. We have stressed, too, the need for designers and metallurgists to get together and consider whether or not better materials are being used than service conditions warrant—a luxury we can no longer afford. This is not to suggest that unreasonable risks should be taken, or the remedy might prove to be no more than an aggravation of the problem.

Similar co-operation on yet another aspect of the supply problem would also seem to hold out a possibility of fruitful results. Once material is declared scrap, the sooner it finds its way to the reclamation plant the better, and as far as scrap in the form of obsolete or broken machinery is concerned, there is little more to be said or done about it. There are, however, other types of scrap arising during manufacture—some of them worthy of further consideration. Such scrap can arise at any stage, from the original ingot or casting to the finish machined component, and a certain amount, in the form of ingot discard, casting risers and runners shearings and turnings, etc., is unavoidable, but a close watch should be kept on this class to ensure that it does not exceed reasonable proportions. It is when we come to consider the material which becomes scrap by reason of its failure to comply with requirements, that there is scope for that practical and realistic attitude of mind which was responsible for the adoption by the American automobile industry of the "kerb-side" finish, to which reference was made in the recently published Productivity Report on Metal Finishing.

This standard is based on the view that the finish on a motor-car should be durable and should possess a satisfactory appearance from the normal viewing range (i.e., 6–12 ft.), and the Team point out that insistence on high standards of finish based on close visual inspection is a considerable handicap where productivity is concerned. In much the same way, insistence on *unnecessarily* high standards, can prove a wasteful drain on raw materials.

Recent years have seen great strides made in the examination of materials by non-destructive testing methods, and the inspector has more means at his disposal than ever for finding causes for rejection. This situation has proved to be something of a mixed blessing, as each method developed demands a new technique in interpretation and new standards of tolerance, for—depending on the sensitivity of the method—imperfections of a minor nature may be found along with the more serious defects. During the early years of the war, a great increase in the use of magnetic particle testing on a mass-production basis took place, and there is little doubt that, in the early stages, really defective components were accompanied to the scrap heap by many which would have given satisfactory service. Most certainly, these improved methods of inspection are proving invaluable, but they have brought with them added responsibilities in the way of interpretation.

Too often the inspection department's motto seems to have been based on Petain's call at Verdun—"They shall not pass"—and a high proportion of rejects is taken as evidence of a high standard of inspection, when it may really be nothing of the sort. The highest standard of inspection is, undoubtedly, that whereby the only material rejected is that which would cause failure, or at least difficulties, in service. Particularly where high stakes are involved, there is something to be said for the attitude: "If in doubt—scrap." That is the safest course to adopt, but it should not provide an excuse for scrapping without consideration. On the other hand, standards should not be established by guesswork, or subsequent service experience is likely to prove unsatisfactory. There is, therefore, much to commend a detailed investigation, by full-scale testing of components, of the effects of the various types of defect found by non-destructive testing, so that it can be established whether or not a particular type of defect matters in the conditions obtaining in service, for that is the real criterion whereby material should be judged.

Distribution Control of Steel

THE new arrangements for the control of the distribution of alloy and non-alloy (carbon) steel, which begin on 4th February, 1952, will be similar to those in operation up to May, 1950, and no person may—with certain exemptions under a Small Quantities Exemption Clause—acquire or use specified types of steel unless he has an authorisation.

The authorisation will permit the consumer to acquire steel direct or to allow his sub-contractors to purchase steel. Any consumer (except a sub-contractor) requiring steel in the controlled forms who has not already stated his requirements to the appropriate Department or to a Regional Office of the Ministry of Supply, should apply at once to the Department which it normally approaches on matters of production. Small consumers—consumers whose requirements are 25 tons or less a quarter including not more than 10 tons of sheets—should apply to the Ministry of Supply Regional Controller. In cases of doubt, application should be made to the Ministry of Supply, Iron and Steel Division, Shell Mex House, Strand, W.C.2.

Present arrangements for allocating sheet steel and tinplate, terne plate and blackplate—which are already subject to distribution control—will remain unaltered. The new Order covers alloy as well as non-alloy steel, but consumers will receive separate authorisations for each type and also for sheet and tinplate.

Iron and steel stockholders will again need licences to acquire controlled types of steel for re-sale. Existing stockholders should have submitted details of their 1950 trading in non-alloy steel to the Iron and Steel Division I.S.1 (a) S.W. Wing, Bush House, Strand, W.C.2. Stockholders who have not already done so and all those dealing in alloy steel should write to this address giving details of their receipt into and sales from stock in 1950.

The Ministry of Supply emphasises that it will—subject to the Small Quantity Exemption provisions—be illegal :—

(*a*) for any firm or person to acquire or use controlled forms of steel unless an appropriate authorisation is held ;

(*b*) to dispose of such steel except to holders of authorisations.

Orders already placed for delivery on or after February 4th will, therefore, require an authorisation. Consumers should, immediately on receipt of their authorisations from Departments, inform their steel suppliers which of their outstanding orders they wish to confirm as being within the tonnage of their authorisations.

Copies of the Order—the Iron and Steel Distribution Order 1951 (S.I. 1951 No. 2006)—may be obtained from H.M. Stationery Office, Price 6d.

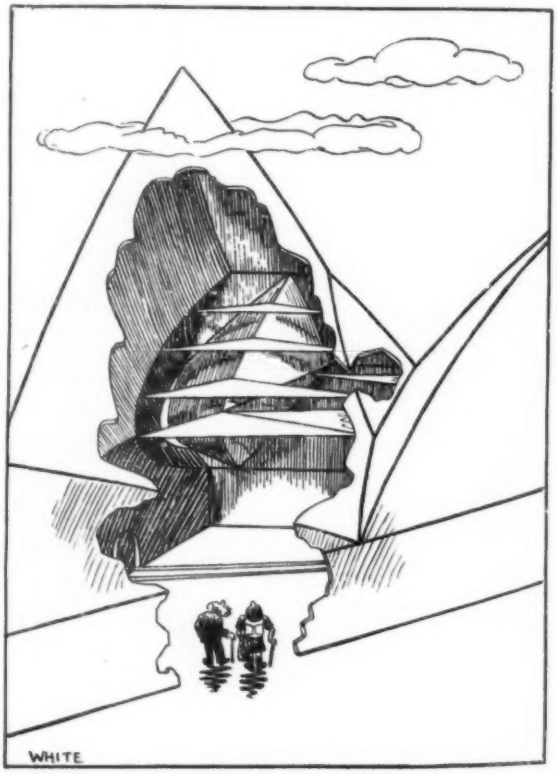
Catalogue Library

THE Engineering Centre, 351, Sauchiehall Street, Glasgow, C.2, has now opened a Catalogue Library which contains the literature of more than 3,000 firms, and also a complete set of British Standards. Frequent additions by the firms concerned, and constant supervision by the Centre's technical staff, ensure that the Library contains only the most recent publications. The facilities offered by this valuable source of information are already being put to good use by consultants, designers, draughtsmen and buyers, as well as by the many overseas visitors who now include a visit to the Centre on their itineraries. It is doubtful whether there is another Catalogue Library in Great Britain which is so readily available to Industry. Manufacturers who have not included the Centre's name on their mailing lists for publicity matter, are invited to do so.

Argonarc Welding Film

A NEW British Oxygen sound and colour film, "The Argonarc Welding Process" was shown for the first time earlier this month. Its purpose is to describe the reasons underlying the development and growth of the process, the first reel dealing descriptively with the equipment and applications of the process in the industrial field, while the second reel is devoted to the welding of aluminium and its alloys. Each reel is arranged so that it tells a complete story and consequently may be shown independently of the other. The two reels together provide an adequate illustration of the various techniques and the scope of the process in industry.

Himalayan Adventure



"OF COURSE IT LOOKS EASY FROM THIS ANGLE," SAID THE OLD MAN. "BUT KEEP OFF THE SLIPPERY SLOPES. YOU MIGHT RUN INTO THE ABOMINABLE SIGMA PHASE."

We are indebted to Dr. James White, Head of the Refractories Department at the University of Sheffield, for permission to publish this drawing. It may not be entirely without value for those who are trying to build up a picture of what happens to the gamma loop in ternary systems.

The First World Metallurgical Congress

A Visitor's Impressions

By K. M. Entwistle, Ph.D., M.Sc.

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Sponsored by the American Society for Metals, and held concurrently with the Annual National Metals Exposition, the first World Metallurgical Congress was held in Detroit in October. In this article, the author presents his personal impressions of the Congress and Exposition.

A.S.M. Headquarters for foreign visitors at the Michigan State Fair Ground where the National Metals Exposition was held.

TO about five hundred visiting metallurgists, a laurel wreath encircling three hands clasping the top of a fluted ingot will always be a symbol of a memorable experience, for this device was designed for the first World Metallurgical Congress, held in Detroit, U.S.A., from October 13th to 19th, 1951.

The author was one of the visitors and wishes to make it clear that, in attempting to give an account of the Congress, many facts worthy of mention may pass unrecorded, since the planned activities were so varied and numerous as to make it impossible for one person to be present at all. This should be regarded therefore as a chronicle of the impressions of one of the visitors.

Study Tours

The Congress was sponsored by the American Society for Metals, and the original germ of the idea smouldered for several years within its genial and efficient Secretary, W. H. Eisenman, until two years ago when time was considered opportune to begin work on the preparations for this great event. At an early stage in the preliminary organisation it was appreciated that if the Congress were to be truly international it would be desirable to arrange for some assistance for European metallurgists who wished to participate. The A.S.M. then approached the Economic Co-operation Administration and suggested the organisation of study tours to precede the Congress. Dr. Zay Jeffries, who was appointed by the A.S.M. to be Director-General of the Congress, spent six weeks in Europe in the early part of the year, publicising the scheme through discussions with those who were to be responsible for selecting the European representatives. A series of eight tours of observation was planned and arranged by the American Society for Metals in the following fields :—

Group 1—Steelmaking and Refining.
Group 2—Non-Ferrous Refining, Rolling and Fabrication.
Group 3—Ferrous Fabrication.
Group 4—Heat Treatment.
Group 5—Welding and Joining.
Group 6—Testing and Inspection.
Group 8—Metallurgical Education.
Group 9—Metallurgical Research.

The programme for each group involved visits to plants, research establishments or educational institutions over a period of four weeks prior to the Congress. On the average, each group made about 16 or 17 visits during that period. The Organisation for European Economic Co-operation organised a Technical Assistance Mission with sections corresponding to the titles of the eight tours and invited Europeans to make application to join one of them. Each group was made responsible for the production of a report covering its visits, emphasising particularly such processes or ideas as could be applied in Europe to contribute to economic development. In addition to the O.E.E.C. members, other overseas visitors joined the tours of observation from countries as widely scattered as Japan and Turkey, Finland and New Zealand. In all, 27 countries (excluding U.S.A. and Canada) were represented by over 170 visitors. A further three tours in :—

Non-Ferrous Heavy Metal Fabrication (Group 7)
Galvanising Techniques .. .. .. (Group 11)
Non-Ferrous Smelting and Refining.. (Group 12)

were organised, in addition, to follow the Congress and, with these, a total of about 300 visiting merallurgists participated. It formed in all the largest technical assistance project which E.C.A. has to date sponsored.

In the first set of visits, of which the author had personal experience, the groups varied in size from 41 (Steelmaking) to 7 (Metallurgical Education) and each

itinerary followed a different route, beginning from New York and moving about roughly within the Washington-Boston-Chicago triangle. All tours converged on Detroit in time for the beginning of the Congress. During the preliminary meetings in New York before the trips set off, each group elected from its O.E.E.C. members its own secretary and chairman to be responsible for co-ordinating reports and reporting activities. During this period, too, the members had the opportunity of observing the very careful thought and planning which the A.S.M. put into the arrangements; everything appeared to have been thought of from baggage labels and medical insurance to transport and detailed notes of the daily programme, contained in a notebook giving, in addition, street maps of all the more important places to be visited. Each group was looked after by an American Tour Manager, who went everywhere with the group and was responsible for transport and similar problems, together with a Tour Escort, a qualified metallurgist who fulfilled a technical liaison role.

American Hospitality

An outstanding feature of these four weeks was the overwhelming hospitality with which the members were received at the places visited. Combined with this, both before and interspersed between visits, receptions and luncheons were arranged by the American Society for Metals. The first of these was a "Welcome to America" luncheon at the Waldorf-Astoria, New York, at which the overseas visitors were welcomed by the Mayor's representative, industrial leaders and World Congress officials. At a "Welcome to the Nation's Capital" luncheon in Washington welcomes were extended by the Government, and education and scientific bodies. On August 20th, 1951, the House of Representatives passed a Joint Resolution introduced to Congress "providing for the recognition and endorsement of the World Metallurgical Congress" and extending from Congress "its official welcome to the foreign metal scientists who will visit major American production centres. . . ." A more light-hearted "Welcome to Cleveland" was given at Sunnimoor Farm, home of the Secretary of A.S.M., where all visitors who were in or near Cleveland at the time were invited by Mr. and Mrs. Eisenman to "an early American party" where "formality will be dropped and fun will be the order of the day." It was!

Overseas visitors were invited during their stay in the United States to attend Local Section or Chapter A.S.M. meetings which coincided with their visits. At the New Jersey Chapter, Walter E. Jominy, President of the A.S.M., talked on Hardenability. Of particular interest was his discussion of the hardenability of boron steels, in view of current work on the possibility of using boron in place of alloying elements which are in short supply. W. Rees of the National Physical Laboratory was invited to contribute to the discussion, during which he stressed the importance of work on steels of controlled composition made by alloying additions to pure iron, a project on which the N.P.L. is at present engaged. Dr. John Chipman addressed a meeting of the Washington Chapter on the "Chemistry of Liquid Steel," during which he outlined the basic thermodynamics of the reactions involved and described recent experimental work carried out at the Massachusetts Institute of Technology in this field. Later in the tour visitors were welcomed to the William Park Woodside Lecture at the Detroit Chapter given by F. P. Zimmerli on "Metallurgy of Mechanical Springs."

The activities planned for the week of the Congress in Detroit were both numerous and diverse; to glance at the full programme for the week was to court mental indigestion. The formal part of the programme comprised three main sections: technical sessions covering a wide range of subjects; meetings for discussion between study tour members and "American opposite numbers"; and the 33rd A.S.M. National Metals Exposition.

Seminar on Interfaces

The technical sessions began with a two-day Seminar on Interfaces, at which American and Canadian workers presented 13 papers summarising the present state of their work in this field. The papers were delivered in four groups: Theoretical Considerations; Interfacial Energies; Movement of Interfaces; and Effects of Interfaces. In the first group, Dr. Convers Herring of the Bell Telephone Laboratories discussed an effect at a free surface in a theoretical calculation of the work function of a clean metal surface. This was considered to be made up of two contributions, one a surface effect and the other a bulk effect. Professor Brooks of Harvard outlined the conception of a grain boundary as an array of edge dislocations, which can be considered to exist in the region of disarray between two neighbouring regions of perfect crystal structure which differ in orientation. From this model it is possible to calculate the total energy of the dislocations and hence derive a figure for the grain boundary energy and its dependence on the relative orientation of the adjacent grains. The results are in good agreement with experiment, at least for small differences of orientation where the dislocation stress fields do not overlap. In a well-delivered and fascinating paper, Dr. C. S. Smith showed how fundamental topological space-filling relations, for example between the number of polygons, edges and corners in a two-dimensional network, could be applied to a study of the shape of metal grains and the phase fields of ternary constitutional diagrams. The conclusions can be illustrated by experiments on two- and three-dimensional bubble froths in which, further, at a reduced pressure, diffusion of gas takes place through the soap films, producing bubble growth. The conclusion from surface tension considerations of the disappearance, in the two-dimensional case, of cells with a small number of sides is substantiated by such experiments and applicable to the case of grain growth in metals. The application of these considerations to metals has been made possible by the development of a technique by Dr. W. M. Williams at the Institute for the Study of Metals, Chicago, for taking stereographic radiographs of aluminium with a small amount of tin which shows up the crystal boundaries.

In the second group of lectures, Dr. Udin of M.I.T. described the technique for surface tension measurement of solid metals at temperatures approaching the melting point. This involves the determination on a wire specimen of the axial load which just balances the surface tension forces tending to reduce its length. The method is applicable to the temperature range in which the resistance to plastic deformation is sufficiently small to be overcome by surface tension effects.

Some beautiful photo-micrographs of aluminium specimens, taken with polarised light, were shown by

The A.S.M. Board of Trustees laying final plans for the World Metallurgical Congress. Those standing are (left to right) A. E. Focke, W. H. Eisenman (Secretary), Evelyn Gardner, J. B. Austin, R. L. Wilson (Vice-President), W. E. Jominy (President) and Dr. John Chipman (President-Elect.). Seated are T. G. Digges, Elmer Gammeter and Dr. McKenzie.

Professor Beck of Notre Dame University to illustrate his paper on grain boundary movement during recrystallisation. Professor Nowick of Yale University discussed the inferences which had been made from internal friction measurements about the mechanical behaviour of crystal boundaries, and outlined his work on coupled relaxations which arise when relaxation centres, that is regions which cannot permanently support a shear stress, overlap.

The A.S.M. Seminar on Interfaces was followed by the opening session of the World Metallurgical Congress, which was devoted to lectures and discussion on world metal resources. James Boyd, administrator for Defence Materials, U.S. Department of the Interior, discussed "Raw Materials for the Metal Industry," "Metals for Defence in the E.C.A. Countries" was covered by a contribution from Pierre Van Der Rest, General Manager of the Belgian Blastfurnaces and Steelworks Association, and Clyde Williams of the Battelle Memorial Institute talked on "Metals for Defence in Non-E.C.A. Countries of the Free World."

Other Technical Sessions

During the rest of the week, regular technical sessions of American metallurgical and allied societies were held concurrently. The A.S.M. held meetings at which a series of over 50 World Congress papers was read, under nine headings: Constitutional Diagrams; Melting and Refining; Diffusion; High Temperature Phases; Mechanical Metallurgy; Physical Metallurgy; High Temperature Alloys; Embrittlement; and Heat Treatment. Visitors were invited to attend the Annual Meeting of the A.S.M.; the large gathering gave some idea of the active support which the Society receives from its 20,000 members. Following the business part of the meeting, C. H. Lorig of the Battelle Memorial Institute delivered the Edward de Mille Campbell Memorial Lecture—"A Metallurgist Looks at Fracture." He pointed out, with the help of a series of beautifully clear simple, coloured slides, how many factors influence the form of fracture and the conditions under which it takes place. This makes it not surprising that no simple and comprehensive theory which will predict fracture in metals appears at present likely. Before the lecture, Walter E. Jominy, the President, paid tribute to the memory of Edward de Mille Campbell, who energetically carried out research work even after being blinded in a laboratory accident during work on the nature of carbides in steel.

Two lecture courses were arranged by A.S.M. and held during the week, one on Residual Stress Measurement by four experts in different branches of the subject, and another by M. A. Grossman of the U.S. Steel Co. on the Principles of Heat Treatment.

At the A.S.M. Annual Banquet, Dr. J. O. Christianson of the School of Agriculture, University of Minnesota, gave an interesting non-metallurgical talk on "Rediscovering America." A presentation of awards was made at the dinner; Paul D. Merica received the A.S.M. Gold Medal, Robert F. Mehl the Sauveur Award and G. A. Price the Medal for Advancement of Research.

Seminar on Dislocations

Parallel with the A.S.M. meetings, the Institute of Metals Division of the American Institute of Mining and Metallurgical Engineers held its annual Fall Meeting in a nearby hotel, and World Congress visitors were invited to the meetings. An outstanding and well attended part of the programme was a Seminar of three lectures on Dislocations in Metals. Dr. Koehler of the University of Illinois, in the unavoidable absence of Frederick Seitz, presented a general introduction covering the nature of dislocations and their properties, introducing the concepts of the Burgers vector and the Shockley half-dislocation. He discussed mechanisms which might give rise to the multiplication of dislocations and a suggested method by which a single pair of screw dislocations may produce multiple slip. Dr. W. T. Read of the Bell Telephone Laboratories described current ideas on the origin of dislocations as a feature of crystal growth. It is suggested that spiral growths observed on carborundum crystals and natural beryl are the result of

crystallisation on the step formed by a surface screw dislocation which forms a nucleus for deposition, and that these spirals constitute direct evidence of the existence of dislocations. He then proceeded to discuss his own work on crystal boundary energies derived from a consideration of the boundary as an array of dislocations. The agreement between theory and experiment in this case is rather surprising in view of the nature of the simplifying assumptions made in the theoretical treatment. Egon Orowan presented a most clear and logical discussion of the application of dislocation theory to mechanical behaviour. A slide of particular interest illustrated the difference between a negative and positive edge dislocation by drawing an analogy to the method of propulsion adopted by a worm and snake, respectively. The worm drags its tail along by propagation of a wave of tension from its head, the snake pushes its head forward by a wave of compression from its tail. This illustrates that the same result is produced in both cases if the "dislocations" are propagated in opposite directions.

The 32nd Annual Meeting of the American Welding Society was held in Detroit during the week and over 70 papers were read at its technical sessions. At the same time the Society for Non-Destructive Testing held its 11th Annual Meeting with eight technical sessions, and the Metals Section of the Special Libraries Association had a two-day meeting. All these meetings were open to overseas visitors.

National Metals Exposition

Concurrently with the meetings, the 33rd Annual National Metal Exposition, suitably enlarged for the occasion, was held in the Michigan State Fair Ground. This is a very spacious and adequate setting for such a show, about eight miles from the centre of the city, where about 400 exhibitors from the U.S. and Europe had stands covering about 6½ acres in seven buildings. A special 15-minute bus service was maintained during the hours of the Metal Show to ferry visitors from the headquarters in the city centre to the Fair Ground. Here, in addition to the exhibits, lecture rooms were available for some of the technical meetings together with a House of Friendship in which visitors could rest, talk and, above all, enjoy that British institution, normally so much neglected in the United States, afternoon tea. This was delightfully served by the wives of past and present A.S.M. officials.

The exhibits covered a fair cross-section of metallurgical and engineering equipment and processes. Two commercially available crystallographic X-ray sets with Geiger-counter spectrometers were on show—the General Electric XRD-3 and the North American Philips. Both use sealed-off tubes which can be replaced in a few minutes. The intensity is derived from the counting rate indicated on a Speedomax recorder, the motion of the chart being synchronised with the angular rotation of the spectro-goniometer.

The R.C.A. Type E.M.T. electron microscope shown is a bench-type model utilising permanent magnet lenses (requiring about 4 lb. of cobalt). It operates at 50 kV. and is normally used with an electronic magnification of 3,000 ×.

A piece of equipment for the cathodic vacuum etching of metals was exhibited. This was deveioped in the Research Department of the Ford Motor Company but is not yet produced in quantity, since its full potentialities have not yet been fully explored. The specimen surface forms the cathode and a discharge of the order of 10 milliamperes at several thousand volts is maintained for about two hours between it and an aluminium anode, in an argon or nitrogen atmosphere at 0·015–0·025 mm. pressure. The etching effect results from preferential evaporation of metal by bombardment of the surface with argon ions. The method promises to be particularly valuable for highly-alloyed heat-resisting alloys and for showing up flow lines in deformed metal.

Direct Reading Spectrometer

The direct reading spectrometer on show will analyse up to 20 elements in a particular class of alloy in two minutes. Each element is determined from the intensity of a strongly characteristic spectral line measured by a photo-multiplier tube. The current from the photo tube is measured from the charge fed to a condenser in a standard interval. Calibration in terms of percentage weight is effected by the use of standard samples. In a more conventional piece of spectrographic equipment interest centred on the source unit which was designed to incorporate a subsidiary spark-gap, in series with the

This year the city of Detroit celebrates the 250th anniversary of its foundation by Antoine de la Mothe Cadillac. At a civic reception to Congress visitors, attended by the Governor of Michigan, M. Bernard Jousset, a member of the French delegation, recalled this early connection with France. In this illustration, M. Jousset (centre) is seen chatting with G. Mennen Williams (left), Governor of Michigan. Between them is Zay Jeffries, Director-General of the Congress, and on M. Jousset's left are Walter E. Jominy, A.S.M. President and Wm. C. Newberg, President of the Dodge Division of the Chrysler Corporation.

main gap, across which a current of air is blown; this produces rapid interruption of the discharge causing it to wander over the surface of the sample and gives an arc more representative of the average composition.

Two stands showed diamond compounds for metallographic polishing and the exhibitors of the "Disa-Electropol" electropolishing apparatus claim "specimen to picture in three minutes"; for steel specimens, throwing a switch performs the etching operation.

In the temperature measurement and recording field, Leeds Northrup shows a Speedomax recorder which will plot a curve relating any two quantities which can be transformed into a D.C. voltage. The relation between the temperature of a wire and its length was plotted in a demonstration experiment. The same firm showed a wide range of controllers, some of the proportional type which adjust the heat supply in proportion to both the rate of temperature change and the deviation from the required temperature.

A vacuum furnace with a hot zone 5 in. × 2½ in. diameter, heated by a single turn tungsten sleeve surrounded by nickel and molybdenum heat shields, is claimed to operate up to 2,000° C. A zirconia sheath is supplied for an immersion thermocouple and the equipment is designed to allow vacuum casting.

The halogen-sensitive leak detector was exhibited in the high-vacuum section. The sensitive element is a platinum diode in which positive anode emission is catalysed by the presence of halogen gas, indicated by a change of anode current on a microammeter. A gas probe issuing Freon gas is moved over suspected leaks which are confirmed by the change of meter reading. The detector operates only in the pressure range 10–0·1 mm. of mercury.

Among the usual types of crack detector, it was of interest to note a supersonic instrument with a separate transmitter and receiver, which is admitted to be more flexible than earlier American designs which favoured a single quartz element used alternatively as transmitter and receiver.

Mechanical Testing Equipment

Competition for markets in mechanical testing equipment, particularly for hardness measurement, appeared from the range of exhibits in this class to be very healthy. An Italian firm demonstrated a Rockwell hardness tester in which the depth of indenter penetration is indicated by the projected image of a graduated scale fixed to the load beam. Methods of autographic recording of stress strain curves, which eliminate the need for direct mechanical connection between cross-head and recording drum, have been a feature for some years now. In one system demonstrated, the movable core of a differential transformer is connected to a Bourdon type pressure element, which measures the applied load on the specimen; load changes cause a displacement of the transformer core and a change of output voltage which is fed to a two-phase servo-motor. This moves the core of a second transformer through a cam to restore electrical balance. The extent of the rotation of the servo-motor in this process is greater than, but proportional to, the core displacement and can, therefore, be used to indicate applied load. In another system, strain measurement is effected from the output voltage developed by a differential transformer type of extensometer, which is fed to an arrangement similar to that previously described. The rotation of the servo-motor is proportional to strain and may be used to rotate a stress-strain recording drum.

Dr. John Chipman and Mr. R. W. Blount (Great Britain) in conversation at the final banquet at the Hotel Statler, Detroit. Mr. Blount is with the Ministry of Education and is responsible for metallurgical education in technical colleges throughout England and Wales.

A European exhibit was the newer type of Schenk Pulsator fatigue tester, incorporating a resonant mechanical vibrating system in the form of a mass attached to helical springs, in place of the flexurally vibrating beam of the earlier design. The Bergsman low-load hardness tester could be compared with the Tukon instrument demonstrated on two stands in the Show.

A wide range of United States and European photomicrographic apparatus was available for inspection; it was of interest to note that the general lay-out of one of the American designs was on the same lines as that of the British Vickers.

There were many stands exhibiting processes and equipment showing considerable ingenuity; an electrostatic method for spraying washing machine bodies, an automatic machine for the control of dimension and hardness of bearings, and an exhibit by a firm of temperature control engineers demonstrating the properties of dished bimetallic discs which flicked continuously between hot and cold plates.

Informal Discussions

A third set of activities arranged during the week in Detroit was a series of meetings between overseas study tour members and American metallurgists working in similar fields. Each study tour had appointed by the A.S.M. a panel of "American opposite numbers" which combined with the visitors for informal discussions, during which questions were raised regarding observations made during the tours, and problems of common interest were debated. The opportunity which these meetings afforded for making personal contacts with Americans interested in similar fields was possibly the most valuable outcome of this part of the programme; indeed of the World Congress as a whole. There can be no doubt that the reports for O.E.E.C. that will be prepared by the study tours will include many useful and valuable findings which can be applied with profit in the metallurgical industry throughout Europe, but, in the minds of those who participated, the less tangible and more human benefit which derives from the meeting of people from many countries with a common interest and purpose will remain the most lasting and valuable

outcome of the World Metallurgical Congress and the preceding tours.

The Congress came to an end at a final banquet in Detroit at which the visiting metallurgists were addressed by Charles E. Wilson, Director of the U.S. Office of Defence Mobilisation. In a broadcast speech he underlined the value of the World Metallurgical Congress and the vital part which metallurgists have to play in world economy. Following Mr. Wilson, leaders of the World Congress made speeches of farewell to the overseas visitors, to which one of their number, in most flattering terms, expressed thanks and appreciation to the many whose efforts had contributed to the success of the venture.

Acknowledgment

The author wishes to acknowledge his indebtedness to the American Society for Metals for providing the photographs used to illustrate this article.

Preheating Tinplate Rolls*

DURING normal working, the cast iron rolls used in the tinplate industry operate at a temperature of approximately 400° C., being hottest at the centre of the working surface with the temperature decreasing towards each end due largely to heat losses at the necks. It is, however, general practice in the industry to commence rolling at a temperature of 250 to 300° C., the increase in temperature to 400° C. taking place during the subsequent rolling.

Thermal expansion of the rolls varies throughout their length in proportion to the temperature gradient. In order that the rolls shall have a uniform gap when hot, they are turned with a slight curvature, so that when cold, they are of a greater diameter at the ends than at the middle.

After a shut-down, either at week-ends or for mechanical breakdown, preheating of the rolls is necessary before sheet of consistent gauge can be produced. This preheating can be accomplished by rolling "wasters," i.e., low quality material, which results in waste of labour, material, and production time, or an alternative is to use preheating burners. These are applied during the period before commencing work and during any shut-down period during which it is desired to maintain roll temperature. The efficiency of the preheating process is affected by the following factors:—

1. Rapid preheating must be avoided since it produces heavy stresses in the rolls due to uneven temperatures between the outside and inside of the roll. This tends to cause fracture of the rolls under load, resulting in an expensive replacement.
2. To increase the efficiency of the process, some means of reducing radiation and convection heat losses from the rolls is desirable. This, however, is not practicable, since any equipment used must be of robust construction to withstand the heavy-handed treatment which it is likely to receive in day to day working. Any type of insulated cover, if robust enough, would be too heavy to handle, especially when hot; if a light construction were used it would not survive for long.
3. The burner equipment used must be of robust construction so that rough handling will not readily result in damage or maladjustment. It must be arranged so that it can quickly and easily be placed in position when required and readily removed when work is commenced or resumed.

Burner Unit

To satisfy these conditions, and to test the suitability of Hypact burners against the conventional method of using bar burners incorporating gas and air under pressure, an experimental unit was made, using seven No. 60 Hypact burners. Temperatures were estimated by means of "Tempilstiks," the range of these available being 200, 250, 300, 400 and 500° F. The burner used consisted of a 2 in. diameter float into which was welded ¾-in. B.S.P. sockets in a single row at 5 in. centres. Into each socket was screwed an 8 in. length of ¾-in. barrel; each fitted with a No. 60 Hypact burner. These burners were arranged so that the wide planes of the flames were parallel to the axis of the float. The effect, when ignited, was to produce a thin sheet of flame directed horizontally towards a point approximately 3 in. below the "nip" of the rolls. The burner was so placed that the flames struck the bottom roll, so that the "dead space" of each flame was about ½-in. from the point of contact with the roll. The burners were connected by 1¼ in. diameter armoured hose.

Performance

During the first test, caution was necessary in case too much heat was applied to the roll, but from the information obtained it was obvious that "full out" operation of the burner from the commencement of the test would not be harmful; this procedure was adopted during the second test. The gas supplied was of calorific value of 350 B.Th.U. per cu.ft. and with a specific gravity of approximately 0·6 (air = 1). In the first test, the rolls revolved away from the burner towards the "nip." In the second test, for greater convenience, the burner was placed in the opposite side of the rolls so that the rotation was, in effect, reversed. The working portion of the rolls being 34 in. long × 24 in. dia.; the total weight of the pair was 6 tons.

Summary of Test Results

Weight of rolls	6 tons
Final temperature	430° F. (223° C.)
Duration of heating	6 hours 35 min.
Gas rate	518 cu.ft. per hr.
Total gas consumed	3,415 cu.ft.
Rise in temperature	330° F. (186° C.)
Heat absorbed by rolls (Sp. Ht. of C.I. = 0·122)	540,000 B.Th.U.
Heat input (350 C.V. gas)	1,195,000 B.Th.U.
Burner pressure	2·5 in. w.g.
Apparent thermal efficiency	45·2%

NOTE.—This is not a true thermal efficiency since:—
(a) the necks of the rolls are at a lower temperature than the main body.
(b) The surface temperature is higher than the centre of the rolls during the heating up.
The figure does, however, form a useful basis for comparison with other similar applications.

*Reproduced from Report No. 51/1/86 of the Industrial Gas Development Committee of the Gas Council (Information supplied by the Wales Gas Board).

Conclusions

From the curves obtained it is evident that a heating period of 9½ hours at about 530 cu.ft. per hour would be needed to attain a temperature of 250° C. (temperature at start = 38° C.), and that, in view of the falling off of the rate of temperature rise around this temperature, it would be difficult and expensive to obtain any further increase in temperature. A more rapid rise in temperature and a higher ultimate temperature could be obtained by applying a pair of burner units to each pair of rolls, the burners to be placed in similar positions on each side of the "nip."

It is essential when using such a burner assembly that it be accurately located with respect to the rolls. The optimum position of the burners for heating rolls of about 24 in. diameter appears to be that for firing horizontally towards the rolls in a plane approximately 3 in. (vertically) below the "nip," with the flames to strike the lower roll so that the "dead space" in the flame is unbroken.

Cold-Forming Steel Sections

By R. Saxton

Some of the factors concerned in the production of steel sections of small cross-sectional area are considered, and brief reference is made to the hot-drawing process.

THE efficient reduction of cylindrical form steels to various shapes and segments by cold processing, necessitates material of good structural quality and plasticity. Even flow of metal under pressure during the first pass or reduction is rarely possible, due to the variable deformation essential to shaping, and plasticity must be such as to permit irregular pressure without detriment to structural formation.

Sections of various types are produced in all qualities of steels, and, where size and formation permit, general practice is to roll hot and shape in the heated rod a facsimile of the desired section in a larger size. This process, which is only applicable to large or heavy segments, reduces the number of passes in cold working, and cuts out one or more intermediate annealings. Hot shaping of small segments is impracticable, due to the rapid cooling off of the material during the process.

Unlike reduction in cylindrical formation, in which, due to the uniform incidence of applied forces, homogeneity is more easily achieved, the application of deformation pressure to sectional units results in a crystalline structure of irregular form, due to variations in applied pressure.

Elimination of this defect is achieved by suitable heat treatment, but as the finished section is required, in almost all cases, to possess a high tensile strength, its use in the as-annealed condition is not practicable, and the reduction must be so arranged as to furnish, in the final pass, a deformation pressure which is uniform over the entire area.

Due to the small cross-sectional area of many segments, their fabrication to a shape favourable to economic production by hot rolling is not a practicable proposition, which necessitates the majority being cold worked to shape from the cylindrical form. Reduction to a shape from a round often necessitates the elimination of fully two thirds of the cross-sectional area of the metal mass, before the desired section and size are attained, a process necessitating frequent heat treatment to eliminate detrimental stress effects.

In the shaping of irregular segments, greater pressure is exerted at certain points, and this leads to heavier flow and grain elongation at these points, with resultant increased internal stress. Metal flow in cold deformation is a complex process, based on block slip along certain planes within the crystals, known as slip planes, accompanied by a tendency of the blocks of atoms to rotate in such a way that the slip plane direction approaches that of the axis of tension. In general, a slab or billet consists of innumerable crystalline grains of random orientation, and as the applied load increases elastic deformation first takes place. This deformation is assumed to consist of distortion of the atomic lattice, without slip. At the elastic limit stress, plastic deformation commences by slip within the grains most suitably oriented, and progresses throughout the structure.

The first consideration in preparation for cold working is to determine the relative area of starting size and finished segment. It is easy to miscalculate, particularly with intricate sections necessitating the formation of sharp edges, and material in the past has often had to be scrapped due to failure to allow sufficient area for efficient finishing of projections or edges.

In the conversion of cylindrical material to square, the rod or wire in the first pass makes contact with the reduction zone at four small points only, the material which will form the corners later passing through without contact. Irregular reduction in this pass permits heavy deformation at the points of contact, and in fabricating mild steel qualities it is possible to reduce 40% deeper than the normal reduction of round stock in similar quality material, in which reduction is accomplished by circumferential pressure.

In passes prior to finishing, sharp edges at corners are avoided in fabricating square section, as these are detrimental to further reduction and exert a cutting action on the die at the points contacted, with resultant detriment to die and material. Only in the last, or finishing, pass are the sharp edges incorporated, and this is the practice with all sections in which such edges are essential.

Locking segments, as employed in the construction of locked-coil wire ropes, are one of the most difficult sections to cold work from cylindrical material, as it is essential that each segment, when incorporated in the rope, locks with neighbouring units with the minimum spacing between. Fig. 1 illustrates the cross-section of a locked coil winding rope with segments in the outer layer.

To attain the tensile properties essential in these ropes, combined with the necessary flexibility, the practice is to anneal fully in rod form and after each pass, with later a special continuous normalising process, known as "patenting." This is employed prior to finishing passes. Standard patenting treatment for rods or wire prepara-

tory to cold working consists in heating the material to a temperature between 950° and 1,050° C., and allowing it to cool fairly rapidly in air or molten lead. The object of this treatment is to produce a microstructure favourable to reduction by cold working. The effect of the high temperature is to reduce the heterogeneity of the material, eliminate defects imparted by previous work, and furnish a structure of fairly large homogeneous grains of austenite. When cooled fairly rapidly, as in air or in a lead bath, the austenite breaks down into a fine structure in which the carbide is uniformly distributed in the form of fine pearlite. This structure (sorbite) is the condition in which steel is most suitable for cold work, as it possesses both ductility and strength.

A defect incidental to this form of heat treatment, yet one which is otherwise beneficial, is the decarburisation of surface layers. Loss of carbon results in a proportionate increase of ferrite, and as ferrite is milder than sorbite it acts as a lubricant between the harder steel and die. The amount of decarburisation varies with the different qualities, but, in general, is so slight as to be almost invisible under the microscope.

In forming segments as illustrated in the outer layer of locked-coil ropes, the first pass or draft consists in the sinking or formation of the two grooves, essential as foundation of the locking area. Correct sinking and depth are important, too light a pass necessitating heavy reduction in the second pass. Many attempts have been made to form this shape by hot rolling to a suitable size for the finishing operation after normalising. As section at this stage is much too small to produce in the hot rod mill, the majority of experiments have consisted of attempts to shape the material by passing it through shaped rolls on emerging from the patenting furnace, but in practically all attempts, deformation has been so severe as to render the material unsuitable for further processing.

In some cases, an enlarged section may first be formed in the hot rolling mill. For example, the section shown in Fig. 2b is formed in this way and finished in two passes with one intermediate anneal. This section is employed fairly extensively in conjunction with cylindrical units as half-lock in half-lock aerial rope construction, as can be seen in Fig. 2a. During cold working the grooves are shaped to permit insertion of a fixed-diameter cylindrical unit between two grooves. This method of rope fabrication exerts a semi-lock action on the outer layer of units, and prevents a fractured wire coming out of lock.

Hot-Drawing Steel Wire

During the past few years, experiments have been conducted with a view to developing methods of hot drawing steel wire. This work has been undertaken primarily for the purpose of furnishing higher output

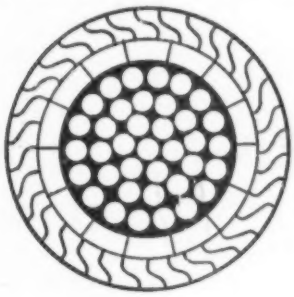
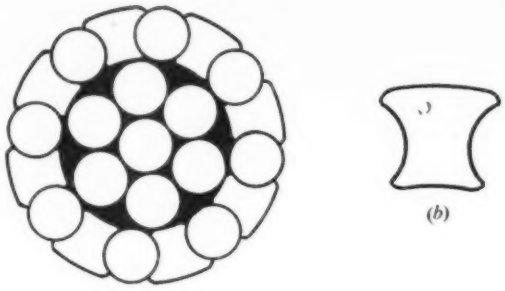


Fig. 1.—Cross-section of locked-coil winding rope with segments in the outer layer.



(a) (b)

Fig. 2.—(a) Cross-section of half-lock aerial rope with segments and units in position in the outer layer. (b) Enlarged segment.

with decrease in production costs, particularly with high carbon, high speed and similar speciality steels, and also to eliminate the hazards and expense of repeated annealings incidental to the production of these qualities.

The following commercial technique and practices are the result of metallurgical studies undertaken to evolve a method which would not involve such defects as surface decarburisation and those imparted by acid cleaning, and it is claimed that, by the use of this method in the drawing of high-speed steels, a product of improved quality is obtained, with superior structural condition and greater toughness.

Plant or drawing equipment consists of a one-pass draw block with a rotating variable speed drum ; a gas-fired lead bath in the form of a tube with goose neck ends ; automatic lubrication of the wire between the exit end of lead bath and the die entrance by a graphite spray ; and the usual pay-off reel or swift. Dies are of tantalum or tungsten carbide.

The first process is a full anneal of the rod or wire in a protective atmosphere, after which the coil is transferred to the hot draw plant. The initial, or what is termed the roughing pass, is followed by further hot draw passes, and the number of successive reductions can be as high as eight or ten, the actual number being dependent on required finishing size or diameter. It will be noted that by employing this process the essential intermediate annealing and acid cleaning required in cold drawing are eliminated.

The reduction per pass, even with compositions or alloys which normally do not lend themselves to cold drawing, equal or exceed those regularly employed in the cold plastic deformation of the milder quality steels, and with high-carbon, high-chromium, and high-speed steels, single pass reductions of 25% are not uncommon.

In production, the wire during travel through lead bath is heated to a temperature exceeding 500° C., but this is decreased as it enters the draw die to within 330°–360° C., and the die is heated to a similar temperature to prevent material seizing during travel through reduction zone.

Though the successful development of this process introduces a new method favourable to the economic production of high speed and similar qualities, it is questionable whether it can prove a serious competitor to the present system of cold drawing mild steel by multiple drafting, but in the reduction and forming of many high-carbon shapes and sections, high speed and similar qualities, it should prove an asset in the elimination of the time lag, due, with present methods, to the large number of annealings and acid cleanings.

Overcoming "Orange Peeling" on Stretch-Pressed Aluminium Alloy Aircraft Skinning

By G. W. Weeks, A.I.M.

(Aircraft Division Chief Metallurgist, the de Havilland Aircraft Co., Ltd.)

The manufacture of difficult stretch-pressings in Alclad aluminium alloy sheet by established methods may result in gross "orange peeling" of the metal surface. The difficulty is overcome by employing a sub-critical annealing treatment.

MANY parts of the modern all-metal aircraft skin structure are manufactured by the stretch-pressing of aluminium alloy sheet. One such component which is of particularly difficult shape is the de Havilland Vampire fin skin which is produced as a single pressing. The pressing is made from 22 s.w.g. D.T.D. 610B Alclad material, and is produced by folding a sheet over the stretcher tool and stretch-pressing in suitable stages, including inter-stage annealing treatments. The component has an appreciable double curvature and it is not possible to manufacture by stretch-pressing the freshly solution-treated material; neither is it possible to complete the forming after carrying out a single annealing treatment. Inter-stage annealing followed by final solution treatment and stretching to finished shape has been found essential.

Initial Manufacturing Process

In the earlier days of the manufacture of the Vampire fin skins, a manufacturing sequence based on previous experience was evolved. This sequence comprised:

(*a*) Super anneal;
(*b*) Fold sheet over a 2-in. diameter mandril, followed by a 1st stage stretching;
(*c*) Super anneal;
(*d*) 2nd stage stretch-pressing;
(*e*) Solution treat;
(*f*) 3rd stage stretch-pressing;
(*g*) 4th stage stretch-pressing.

The super-annealing treatment in use involved heating the sheet to 425° C. and then cooling slowly, at 15° C. per hour, down to 275° C., followed by withdrawal of the sheet stock from the furnace and cooling. This treatment gives a very ductile material and the following mechanical strength figures are typical of D.T.D.610 material so treated:

0·1% Proof stress	5·25 tons/sq.in.
Ultimate tensile stress ..	12·5 tons/sq.in.
Elongation	17·0%

However, it was found in practice that this manufacturing method was not entirely satisfactory. Whilst some pressings were produced with a good surface, many had, to a greater or lesser degree, an "orange-peel" surface over a wide area. A typical bad case is illustrated in Figs. 1 (×$\frac{1}{10}$) and 2 (×2). Not only was the surface finish undesirable from the aerodynamic and fatigue resistance points of view, but mechanical strength tests confirmed that the material of the skinning was below specification requirements for D.T.D.610B. Resort to a precipitation treatment of five hours at 175° C. effected an improvement in properties, but in many cases the material still did not meet the minimum specification requirements, and in any case this had no effect on surface finish. Table I gives typical mechanical strength test results for pieces cut from an "orange-peeled" stretch-pressing.

Metallurgically the incidence of varying degrees of "orange peeling" was not very mysterious. The phenomenon was quite evidently associated with the

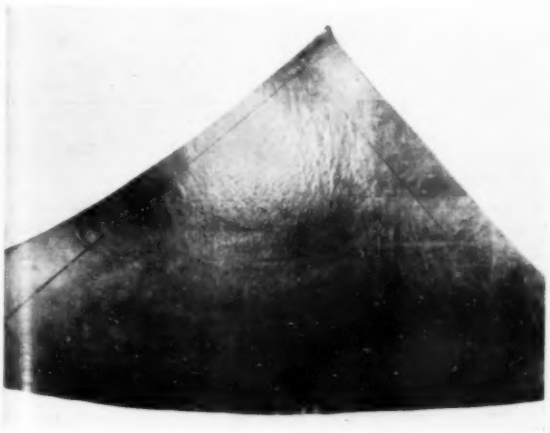


Fig. 1.—"Orange-peel" surface on Alclad pressing. ×$\frac{1}{10}$

Fig.2.—"Orange-peel" surface on Alclad pressing. ×2

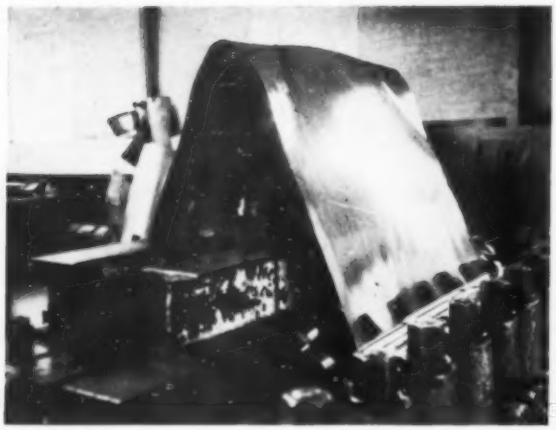


Fig. 3.—The first stage of stretch-pressing.

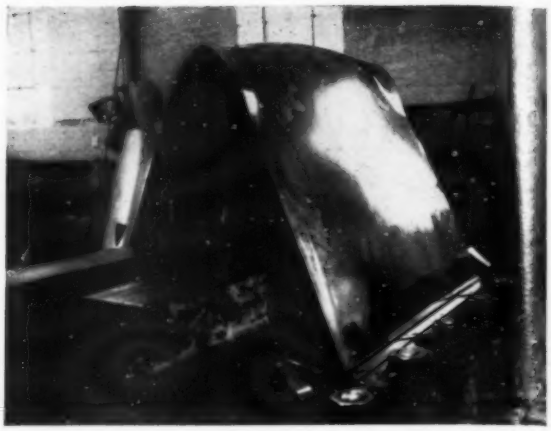


Fig. 4.—The second stage of stretch-pressing.

amount of mechanical cold work employed in forming the pieces and the employment of an annealing temperature well above the recrystallisation temperature. Also the actual rate of heating and cooling the critically-cold-worked material would certainly influence crystal grain growth. It appeared that it might be possible to control, or even eliminate, the incidence of "orange-peel" effect, either by controlling the amount of cold work, or annealing conditions, or both.

Laboratory Investigation

As a preliminary, a programme of work was carried through in the laboratories to establish the amount of cold work necessary to produce the "orange-peel" effect and to confirm its deleterious effect on mechanical strength properties.

The programme of work was fairly extensive and involved the investigation of the effect of various percentages of plastic strain on the incidence of "orange peeling." Material was tested by straining in the "as-received," "freshly solutionised" and "super-annealed" conditions respectively. Triplicate tensile test pieces were strained to various extents and then re-solution treated and strained to "orange-peel" point; or to failure if no "orange peeling" occurred.

Summarising the work done, it was established that "orange-peel" effect only occurred on super-annealed material and only if that material had been subjected to a plastic deformation involving 10% stretch or more whilst in the super-annealed condition. The "orange-peel" effect occurred on stretching after the re-solution treatment. It was confirmed that the "orange-peel" effect was indicative of low mechanical strength properties. Fairly obviously, if the super-annealing operation could be eliminated, then there was every hope of producing satisfactory components.

With this thought in mind, a programme of work was put in hand with the object of investigating the effect of various annealing temperatures on the mechanical properties of the material. The object was to secure adequate softening of the material at a sub-critical temperature, so that forming could be achieved without subsequent "orange peeling." Table II details the mechanical strength properties recorded after annealing for one hour at various temperatures. From a consideration of these results, it was clear that an annealing temperature between 275° and 325° C. might well give adequate ductility, though the upper end of the range might give trouble by crystal grain growth.

The last stage in the programme of laboratory work involved investigation to ascertain whether or not use of

TABLE I.—MECHANICAL STRENGTH TEST RESULTS FROM AN "ORANGE-PEELED" STRETCH-PRESSING.

	0·1% P.S. tons/sq.in.	U.T.S. tons/sq.in.	Elongation %
No. 1 as D.T.D.610	14·2	22·6	9·5
No. 2 as D.T.D.610	14·3	22·8	12·0
D.T.D.610 minimum requirements ..	15·0	25·0	—
No. 1 as D.T.D.546B	22·0	25·8	4·5
No. 2 as D.T.D.546B	22·7	25·6	4·5
D.T.D.546B minimum requirements..	21·0	27·0	—

TABLE II.—MECHANICAL PROPERTIES OF D.T.D.610B AFTER VARIOUS ANNEALING TREATMENTS.

Annealing Temperature	0·1% P.S. tons/sq.in.	U.T.S. tons/sq.in.	Elongation %
200° C.	23·1	26·6	6·0
225° C.	21·5	26·2	6·0
250° C.	17·1	23·7	5·5
275° C.	11·7	19·9	12·5
300° C.	9·53	17·0	11·5
325° C.	7·32	15·85	14·5
350° C.	6·92	16·0	15·5
375° C.	6·68	15·6	18·5
400° C.	7·12	17·0	16·0

TABLE III.—MECHANICAL STRENGTH PROPERTIES AND INCIDENCE OF "ORANGE PEEL" AFTER STRETCHING 10% AFTER VARIOUS ANNEALING TREATMENTS, FOLLOWED BY RE-SOLUTION TREATMENT AND NATURAL AGEING.

Annealing Temperature	Test Piece Identity	0·1% P.S. tons/sq.in.	U.T.S. tons/sq.in.	Elongation %	Remarks
275° C.	1	18·45	30·1	14	
"	2	18·42	28·7	10·5	
"	3	21·5	31·7	7	
300° C.	1	17·8	28·4	13·5	
"	2	18·0	29·3	17	
"	3	19·25	29·2	9	
*325° C.	1	15·3	27·8	16·5	Slight orange peeling nr. fracture
"	2	14·95	28·2	16	
"	3	17·05	28·8	16·5	
Spec. D.T.D.610B requirements:		15 min.	25 min.	Not specified	

* It was impossible to obtain a 10% stretch after annealing these test pieces, the actual stretch obtained being 8%, 9% and 9% respectively.

Fig. 5.—The third stage of stretch-pressing.

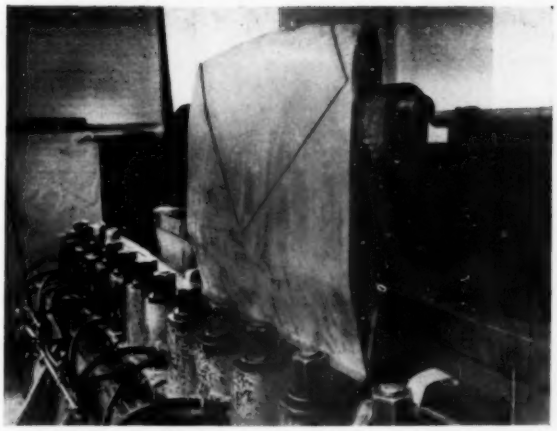


Fig. 6.—The fourth and final stage of stretch-pressing.

a lower annealing temperature would eliminate the "orange-peeling" phenomena. To check this point, triplicate tensile test pieces were annealed for one hour at various temperatures over the range 275° to 325° C. The test pieces were then given a 10% stretch whilst in the annealed condition.

Finally the test pieces were re-solution treated, allowed to age naturally and the pieces strained to observe the incidence of "orange peeling" and to record the mechanical strength properties. The test results of this last series of tests are given in Table III.

Production Tests

On consideration of the information obtained as a result of the work already described, it was decided that an inter-stage annealing treatment of 275° C. for one hour should be tried out in practice, and as a preliminary, ten pairs of stretch-pressings were processed under the new heat-treatment condition.

The manufacturing sequence was the same as that detailed previously but in place of each super-annealing operation an annealing treatment of one hour at 275° C. was employed. These ten pairs of fin skins were processed quite satisfactorily without any development of "orange peeling." It was found, however, that because of the rather less ductile sheet, the degree of deformation which could be applied at the first and second stages of stretch-pressing was slightly less (due to the risk of fracture), but this was overcome by increasing the deformation during the last two stages.

Fig. 3 illustrates the first stage of stretch-pressing. Deformation is carried out to the limits indicated by the chalk lines on the top of the fold. Figs. 4, 5 and 6 illustrate the second, third and fourth stages of stretch-pressing; the outline of the finished fin skin is seen marked out on the pressing in Fig. 6.

It may be of interest to note that the last two stages of stretch-pressing are carried out following one solution-treatment operation. The quenched blanks are stored in a refrigerator and withdrawn in sufficiently small batches for the third stage of stretch-pressing to be carried out without material natural ageing. These blanks are then returned to the refrigerator and later withdrawn to carry out the fourth and final stretch-pressing after the second tool has been set up in the machine.

Since the completion of the first small batch of pressings, production quantities of these pieces have been successfully manufactured with the complete elimination of "orange peeling."

Acknowledgments

The author wishes to make acknowledgment to the de Havilland Aircraft Co., Ltd. for permission to publish this article and to Miss Barbara Bentley, B.Sc., of the Metallurgical Laboratories, who carried out the greater part of the investigational work involved.

High Purity Materials

Johnson Matthey Products Commended

TRIBUTE is paid to the quality of Johnson Matthey pure metals and compounds in a publication recently issued by the Technical Information Division of the United States Atomic Energy Commission at Oak Ridge, Tennessee. This publication, entitled "Availability and Sources of the 96 Elements in High Purity Forms (URCL—266)," contains a foreword in which the author—Elwin H. Covey, of the University of California—stresses the great importance of such materials in modern research. The following is an extract from the foreword: "Compounds and metals are listed in order of decreasing purity. Johnson, Matthey and Co. generally heads each list. This company's products serve as reference points to indicate the most reliable high purity material generally available. Other materials and sources may be judged relative to this company with its products as standards."

Move to Larger Premises

FINA PETROLEUM PRODUCTS LTD., announce that, due to the rapid expansion of their business, and the corresponding increase of their Head Office administrative staff, it has become necessary to remove to more commodious premises. Accordingly, after more than three years at their Mayfair address, they have now secured a seven-floored block of offices in Westminster which will allow greater convenience and comfort in dealing with the Company's growing requirements. The new Head Office address is: Fina Petroleum Products Ltd., 25, Victoria Street (South Block), London, S.W.1. Telephone: Abbey 7822.

Notes on the Metallising of Plastics

By D. McPherson

Although the coating of plastics with metallic deposits has found its main application in the production of novelties, the development of suitable coatings would open up important possibilities in more serious fields. An outline of the processes in use is presented here, together with an indication of developments in progress.

IN recent years a great deal of attention has been devoted to the metallic finishing of plastics. The trend is significant in that it reflects, on the one hand the importance of novelty in certain classes of synthetic commodities, and on the other, the growing realisation that the successful development of true composite structures of metal and plastic might exert a profound influence on many industrial methods. The fact that, to date, more attention appears to have been devoted to frivolous ends, may reflect, one, a consumer resistance to mass produced plastics, and two, that a suitable process, versatile and robust enough to accommodate industrial requirements, has not been available. There seems little doubt that easy acquisition and overproductivity are two factors encouraging consumer-resistance to many non-utilitarian plastics. Novelty, on which such articles are so dependent, can be a peculiar task master, and having exhausted the resources of colour, hygiene, lightness of weight, etc., any alternative, such as a metallic finish, is eagerly seized upon. Thus we have seen ingenious applications of physics built into complicated and expensive vacuum deposition plants in order to provide an eye-catching glitter on hair slides for the multiple store. Thought provoking as the latter may be, it must be said in fairness that a genuine demand has also been present for an industrially efficient metallo-plastic, but it is only recently that the metallurgist-cum-plastic technicians have solved in part the problem of "alloying" metal to plastic surfaces. Because of this, it may be of interest to examine the limitations of available processes in the hope that thereby a better understanding of the background to the problem will result.

Vacuum Deposition

Metallurgically, the principles of vacuum deposition are well established, and have received adequate attention in the technical press. Its application to plastics has followed fairly orthodox lines, with modifications in vacuum technique introduced to accommodate particular chemical conditions.

Much as it might help, it is not possible, however, to compare the vacuum deposition of plastics with any metallurgical process designed for a similar purpose, *i.e.*, the creation of a duplex structure at the surface. Indeed, it is stretching the meaning of the word 'metallising" somewhat to include a lacquer sandwich (the meat of which is the vacuum deposited film of metal) with processes so described.

The quality of plastic mouldings from the viewpoint of surface finish is very often deceptively poor, and in order to obtain "brilliant" vacuum deposited films of metal the surface should be as near as possible to a mirror finish. Super-finished surfaces are, oddly enough, often more difficult and costly to obtain on plastics than on metal. For this reason, the surface tension properties of synthetic lacquers have been exploited to provide a lustrous coating on which bright vacuum deposits can be conveniently developed. In order to prevent destruction of the micro-film, it is necessary to protect it with a further lacquer coat. Thus the vacuum deposited film can be said to exist remote from its host material, and its life can only be the chemical and abrasive life of the lacquer film by whose courtesy it persists. Although many attractive effects can be obtained by such methods, under conditions of moderate abrasion its life is short, and its application thereby seriously limited.

It is, perhaps, not quite fair to describe vacuum deposition on plastics as a laborious way of producing a "catchpenny" effect, but many current uses of it do little to discourage this cynical remark. Back reflection through transparent plastics, such as the acrylics, might be singled out for special mention in that it represents a much more permanent proposition, but it is difficult to see how deposition on exposed surfaces can make contributions other than in the decorative and reflective field.

"Shiny Spray" Processes

Currently listed among metallising processes for plastic materials are those conveniently described as "shiny spray." Such methods, which utilise the chemical reduction of ammoniacal silver nitrate solutions supplied under pressure through spray guns, may be regarded as less technically competent devices to produce the vacuum deposited effect. If there are grounds for questioning the inclusion of vacuum deposition as a metallising process, the same must apply to the "shiny spray" treatment, as the film of silver reduced at the work face also occurs in the form of a lacquer sandwich, and durability is, likewise, a property of the lacquer rather than a metal.

The chemical reduction of silver is a comparatively simple reaction, but in commercial practice it has been found to be difficult to control. It is now well over a century since J. V. Liebig's first discovery of chemical mirrors and, in spite of the many recipes since produced, wet silvering is still regarded by competent people as being far more of an art than it is a science. In the United States, "shiny spray" methods are being exploited to provide an attractive glitter to cheap costume jewellery and moulded toys, but even in this field it is rapidly being replaced by vacuum deposition because of its unreliability.

Wet Silvering and Electrodeposition on Plastics

Of the methods to be examined, those included in this group are perhaps most entitled to be described as

Continued on page 310.

Trends in Powder Metallurgy

By H. W. Greenwood

(*Powder Metallurgy, Ltd.*)

Considerable progress has been made in powder metallurgy in the post-war years, so much so that there are possibilities of the demand for certain metal powders putting a severe strain on the present production resources. Attention is drawn to a number of directions in which useful trends are taking place, including the powders themselves, infiltration and impregnation processes, high temperature alloys and carbides.

THE attempt to assess progress in any particular field is never a simple matter. There is always so much material, as yet incomplete, and so many directions in which useful trends can be recognised, that no possible finality can be claimed for any conclusion reached. There are always a number of results that can be mentioned, perhaps discussed in some little detail, but the overall picture must be one indicating lines of progress and expansion rather than finite achievements or completed researches. That is very true of Powder Metallurgy to-day. Progress in the post-war years may not have been spectacular, but it has been considerable. Moreover, as in so many other fields, there has been not only an acceleration in progress, but a marked broadening of the front along which advances are being made. Until recently, the volume of production by powder metallurgical methods was not such as to make an appreciable call on raw materials, but to-day there are possibilities in more than one direction that the demand for even so common a material as iron powder may be such as to strain present resources of production, and in other strategic materials the necessity for careful rationing is already perceptible. In considering the trends in powder metallurgy in the last year or so, it may well be useful to bear such matters in mind, and, so far as is possible, to indicate developments valuable both economically and technically.

Metal Powders

A word or two may be said about the much increased demand for sponge iron powder, which grows at such a pace as to give rise to anxiety as to the adequacy of present world supplies. Here there is no possibility of substituting some other easily obtained iron powder. The sponge iron powder used so largely to-day has purity, particle size and physical characteristics with which no other iron powder has so far been able to compete at anywhere near the price at which sponge iron powder can be sold. If one were asked to say in what direction other sources of iron powder might become available, it would have to be in the production of a sponge-like iron powder of high purity, produced by an atomizing process, which would yield a material of sufficient sponginess, and with as low an oxygen content as the present sponge iron. Note that the three governing factors would be purity, particle size, and physical characterisation. These are the factors that must control all metal powders to be used in powder metallurgy, and much of the development work of to-day and tomorrow must concentrate on the study and control of these factors. One valuable service that powder metallurgy may well render lies in enabling source materials to be replaced by substitute materials, this helping to tide over present shortages. As an example, there can be cited the interesting development, by French powder metallurgists, of permanent magnet materials based on sub-micron sized iron powders. The

Components produced by powder metallurgy.

magnets so produced have properties falling between Alnico IV and Alnico V and so can probably replace magnets containing cobalt and nickel for many purposes. In the production of porous bearings of bronze or other copper alloys, electrolytic copper has played an important part and there has been, and is, much activity in the study of the best conditions for the production of electrolytic copper powders. There are already two important plants in operation in Great Britain, and experimental work is in progress for the study and subsequent installation of a third producer in the near future.

A valuable research on the electrodeposition of metal powders has been published in full in Stockholm,[2] and in abstract in this country and in the United States.[3] It deals with the factors responsible for producing powdery or spongy deposits, and covers the electrolysis of copper, iron, nickel, zinc and cadmium. There has been a notable volume of work on factors that can influence the properties of powders, and their behaviour during pressing and sintering, especially one on the influence of oxide on the pressing and sintering of electrolytic copper compact.[4] The protection of metal powders against corrosion from the atmosphere and the stabilizing of powders to enhance their keeping properties are still important problems for which no universal, indeed no really satisfactory solution has as yet been found. The deposition of protective films, either from vapour or liquid media have been the subject of much discussion and many patents have been taken out. A comparatively recent account of work by Russian scientists has been published,[5] in which a moisture-repellant film is deposited on particles of powders produced by electrolytic methods, by adding stabilisers to the wash water used to eliminate electrolyte from the freshly produced powders. Stability and resistance to corrosion for over 12 months was claimed for both aromatic and aliphatic substances used in this way.

Coatings of a different character, and for a wholly different purpose, have been discussed and a number of claims made for which patents have been obtained. Interesting examples were discussed of what have been called Thermochemical Coated Powders.[6] These comprise iron powders (cast iron) heated with pigment-grade oxides or mixed oxides, including copper, copper-lead, and copper-nickel or nickel, and excellent pressing qualities are claimed for them, as well as good physical properties for the sintered compacts.

An English patent[7] has been granted for a coated alloy powder in which coatings of molybdenum or tungsten are claimed to give control of grain boundary material and to influence creep properties. A notable contribution to the surface protection of high melting point metals which, while not dealing solely with powder metallurgical products, is of special significance in this field, has been published as a contribution to the W. C. Heraus G.m.b.H. Centenary Memorial Volume.[8] It contains a valuable bibliography of 40 references as well as describing work on the siliconising of molybdenum in which it is claimed that the protective effect is due to a very thin coating of silica. Methods of procedure are described.

In the carbide field, there has been much investigation and discussion on the influence of grain size, and of mixed crystals as opposed to single crystals of carbides. In particular, the influence of grain size has been emphasized by the greatly increased wear resistance and hardness of tungsten when the grain size of the sintered material is of the order of 0·1 micron as compared to the commoner 1–20 micron particles. This is also a general characteristic and has been found to apply to the metals, carbides and nitrides of molybdenum, tantalum, titanium and boron.

In addition to the attention paid to the individual particle, there has been much discussion of the mechanism of sintering, and a very sound review of the subject with critical discussion of the literature has appeared in this journal.[9] An interesting contribution to the theory of sintering under the title of " A Phenomenological Theory of Sintering " appeared in the Proceedings of the Physical Society,[10] while even more recent communications in the U.S.A. and Switzerland[11, 12, 13] dealing particularly with diffusion during sintering, with reviews of literature as well as some useful contributions to the subject, have been published. Interest has also been manifested in the value of pre-sintering as a means of reducing or eliminating shrinkage during final sintering, and also as a method of producing porous carbide bearings, etc.[14] The subject of pre-sintering deserves attention as a possible contribution to the more economic production of compacts which require to be produced to very close and rigid specifications ; there are also possible applications in the direction of corrosion-resisting grids, screens or sieves, in which spherical particles either of refractory metal or of ceramals, or even ceramics might be usefully employed. Before leaving discussion of metal powders, as such, it may be convenient to mention here an entirely novel procedure that has been reported from Germany, namely the production of metal strips by the cold-rolling of iron powder. While this may not strictly belong to powder metallurgy it has to do with metal powders, to wit, atomised iron powder in the particular case described,[15] and while at the moment the procedure may not appear to be a practical application, it might well develop into something both interesting and valuable.

Infiltration and Impregnation Processes

The use of these procedures is not new to powder metallurgy, for some of the very earliest sintered bodies were infiltrated with mineral oil to aid lubrication of the first bearings produced by powder metallurgy around the 1870's. From then on, the porous bearing was the best-known product of powder metallurgy. After World War II we heard of the development, by Boegehold, for General Motors, of the impregnating of a sintered porous copper-nickel layer on a steel backing with a Babbitt metal or a lead-tin-antimony anti-friction metal : in this way a much improved metal bearing was produced. Rather earlier than this, G.E.C. had produced their heavy metal, perhaps the first example of a metal sintered in the presence of a liquid phase, in this case a copper-nickel impregnation or infiltration in a tungsten skeleton.[16] It was this achievement which really drew attention to the possibilities inherent in the processes of infiltration and impregnation. Pure tungsten, under the best conditions of sintering, remains very porous and extremely brittle. Here, with an addition of only 5% nickel and 2% copper, and using a very fine tungsten powder, a density of almost theoretical value was attained with a tensile strength of over 100,000 p.s.i., after sintering for a comparatively short time at 1,400–1,500° C. By the time the Symposium on Powder Metallurgy was held in London in 1947, much work had been done in America, Austria, Germany and Gt. Britain, particularly on the infiltration of iron skeletons with copper.[17] It was now very clear that by infiltration materials could be produced not only with an extremely wide range of properties, but also capable of surface heat-treatment, of solution heat-treatment, and of precipitation hardening. The general situation, and some of its potentialities was reviewed by the writer,[18] and attention drawn to a particularly valuable paper by Austrian workers[19] covering both possibilities and governing conditions. Not the least interesting item in this paper was the very comprehensive list of examples drawn from most of the metal elements, and including no less than seven metals technically important for infiltration in the sintered porous state, and 12 that could be used as infiltrating metal. Several more metals have been added to both groups since the publication of the paper quoted, and other materials, not metals, can also be used as infiltrants. So far, the most widely applied combination has been the infiltrating of a porous iron compact with copper, or a copper alloy. In the U.S.A. over a million jet-engine compressor blades have been produced by infiltrating iron compacts with a molten copper alloy[20] and such blades have proved not only satisfactory in service, but highly competitive with forged blades, made from 12% chromium steel, which were previously used for the same service. A somewhat similar procedure has been applied to the production of gears.[21]

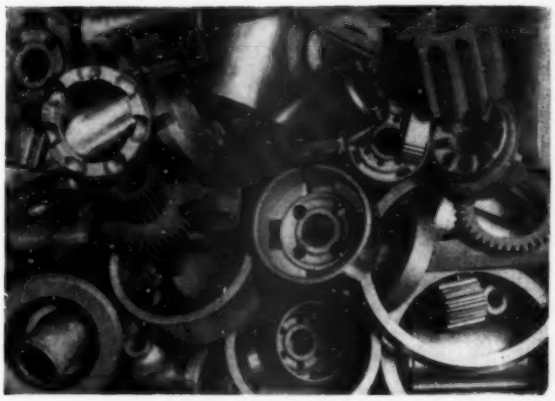
There have been many applications dependent upon the capacity of infiltrated masses for surface hardening, and a single example may be useful. It comprises the production of cutter blades for a food-shredding machine.[22] The blades are produced from copper-impregnated sintered iron. After infiltration, they are pot-carburised, reheated in cyanide and quenched from 845° C. and so a file hard surface is obtained.

Perhaps the most remarkable field for infiltration lies in the highly refractory materials that are of such importance to-day in the development of jet and gas turbine

engines. A glimpse of some of the possibilities is given in a communication by Dr. C. G. Goetzel,[23] outlining a systematic study of the infiltration of skeletons of tungsten and tungsten-chromium alloys with nickel or cobalt base alloys such as Nichrome-V and Hastelloy-C. In such cases the requirements expected of these super-alloys, as they have been called, comprise not only resistance to high temperatures and to corrosion, but also to creep and to fatigue. An interesting suggestion, where resistance to corrosion is required, is the replacing of bronze by sintered iron which has been impregnated with molten enamel.[24] This approaches the field of ceramals, in which ceramic materials are combined with metals to provide resistance to corrosion and high temperatures, and in which a remarkably wide range of combinations have been tried or suggested. It may be convenient to cite a couple of examples here. One is the subject of a British Patent application and comprises blades for turbines slip-cast with one ceramic of high thermal shock resistance, forming the tip, and another of high mechanical strength increasing in thickness towards the base. The root of the sintered blades are metallised with copper, iron or aluminium and then machined to size without affecting the ceramic material.[25] The second example is of a ceramal coating, containing 80% by weight of chromium and 20% frit, which is fired on to a blade consisting of 80% titanium carbide in 20% cobalt by heating for 10 minutes at 1,200° C. in an atmosphere of hydrogen. It is claimed that this permits extended operation of turbine blades at a temperature of 980° C.[26]

High Temperature Alloys

Having already mentioned a number of high temperature resistant materials, it may be as well to discuss the matter further at this point. The metallurgist is fortunate in that there are now available to him the papers presented at the Symposium held at the Iron and Steel Institute, London, in February last.[27] They form a volume of over 300 pages containing no less than 37 communications covering every aspect of the subject. It is not too much to say that this report is the most valuable collection of data and discussion thereof that has appeared to date on this subject. The survey of development, which occupies ten pages, describes in some detail the research work undertaken since 1939 in Gt. Britain, America and Germany on the provision of materials for use in gas turbines. The major part of the Report covers materials other than those produced by powder metallurgy, but the whole will be found indispensable if a balanced view and understanding of the subject is desired. Perhaps the dominant impression produced by a perusal of this report is of the variety and number of major problems that have yet to be solved if the ideal material for turbine blades, and other parts, is to be forthcoming. It is not just a question of producing a first-class refractory material. That is involved, of course, but there is also the question of maintaining physical strength at high temperatures, and that envisages 1,000° C. as by no means the upper limit, and producing at the same time material having high resistance to thermal shock and fatigue, together with resistance to plastic deformation under load at the temperatures, and for the times, essential if an economical performance of the turbine is to be attained. It is also clear that the problems involved in dealing with the gas turbine as used in aircraft are notably different from those which the designer and engineer will encounter in considering land and marine turbines. One conclusion which will inevitably follow consideration of this report will be that the normal metallurgical procedures of melting and casting materials do not offer very great hopes in the production of the materials which will be required if any further notable progress is to be made in providing substances capable of standing up to the much more stringent requirements of tomorrow, and that powder metallurgical techniques alone offer practical possibilities. Another reflection will be that in the solution of the problems confronting us in this particular field, we are obtaining both methods and materials that can be applied in many other directions. It will also be apparent that, particularly in the matter of gas turbine blades, many other materials besides metals will be called upon and will require notable modifications in technique and wide departures from standard metallurgical practice. A valuable review of heat and oxidation resistant sintered materials appeared in April of this year,[28] with 85 references to recent literature. Another valuable review of comparatively recent work is "Review of (American) National Advisory Committee for Aeronautics Research on Materials for Gas Turbine Blades."[29] This contains evaluations of the high-temperature properties of ceramic materials, titanium carbide, boron carbide, and various combinations of these with metal powders, and much useful discussion of bonding strength between ceramics and metals, structural changes after blade failure and cognate matters. By now it will be obvious that carbides will play an important part in the development of gas turbine blades and other heat resisting units and we can, therefore, discuss carbides in further detail.



Courtesy of Sintered Products, Ltd., Sutton-in-Ashfield, Notts

A miscellaneous assortment of products of powder metallurgical processes

Carbides

The carbides in general use to-day have two outstanding properties, their hardness, and their refractoriness. In hardness they come very close to the diamond, the hardest material known, and as far as melting point is concerned they range from boron carbide (m.p. 2,350° C.) to tantalum carbide (m.p. 3,870°C.). In these two properties lies the reason for the close study that has been so notable a feature of the last few years, and which, so far, has not exhausted their possibilities or potentialities. Progress in the last year or so with carbides has been in the

direction of producing compositions that offered advantages not only in hardness, but particularly in toughness, which might be described as resistance to shock and reduced brittleness.

Not so very long ago, the possibility of producing a carbide drill head that could be used on a percussive rock-drilling machine would have seemed remote: now they are in production and successful use. This result is not due to any one outstanding discovery, but has come about by a closer and better knowledge of the effect of particle size on the performance of carbide tools, and of the virtues of the separate carbides. A study of the relations of carbide and cement has helped, and at present much attention is being paid to the mutual solubilities of carbide and cement, and to the role played by solid solutions of one carbide in another. An English paper describing experimental results obtained with what was virtually a solid solution of chromium carbide in titanium carbide, bonded with nickel or cobalt, was published in METALLURGIA last year,[30] and information about titanium carbide with 15% of other known carbides in solid solution and bonded with various alloys is revealed in an Austrian patent.[31] Solid solution of uranium carbide can be incorporated in hard metal compacts with various cobalt, nickel or copper-beryllium alloys according to another Austrian patent.[32] Tantalum-titanium alloys and carbides have been the subject of close study both in America and in Austria, and there has been published in America[33] a valuable paper on hard metals on a tungsten-titanium-tantalum basis, in which phase diagrams of the system at 1,450° and 2,200° C. are published, and attention is drawn to the high transverse rupture strength of the tantalum hard metals which is attributed to the grain growth inhibiting effect of tantalum in the carbide phase.

Another interesting detail in the field of hard metals is their production, so far as complex hard metals are concerned, by a process of infiltration not very dissimilar from that already mentioned under the heading of infiltration. The skeletons to be infiltrated consist of pure carbides of tungsten or titanium, or of mixtures in certain definite proportions of mixed tungsten-titanium and titanium-molybdenum solid solutions. The alloys for infiltration, prepared by powder metallurgical methods, are cobalt with tungsten carbide, nickel-chromium, cobalt-chromium and other mixtures, and details are given of the infiltration methods used. The properties of the products obtained are tabulated in an article published in Austria.[34]

A notable feature of the story of the carbides in the last few years is their ever-increasing use not as components of cutting tools, but as refractories and as abrasion resisting materials in industry. This is true of Gt. Britain as well as of other countries. Many users of carbides are to-day making their own materials, as well as producing the finished dies, containers, machine parts and the like. Not only are large numbers of dies used in wire-drawing, extrusion, and similar operations, but dies for powder metallurgical pressings, both cold and hot, and for many parts which in service are subjected to abrasion, are frequently produced by the users themselves. This has the effect of developing a widely disseminated "know-how" that has been of great value in spreading a knowledge and understanding of the operational handling of the carbides and of the possibilities of powder metallurgy, and has played a useful part in accelerating development and progress.

An excellent example of this has been revealed by the account given before the American Metal Powder Association[35] of the development of gear and small component production by a firm in Yorkshire who have been producers of wire and rod for many years, but who now also use powder metallurgy to make the more complex parts in sintered iron. They make their own dies by hot pressing of tungsten-tantalum carbide, with cobalt as binder, both for the powder metallurgy and the extrusion. It is gratifying to find an English manufacturer not only studying and applying the latest methods of manufacture, but lecturing on them before an American Association.

It may be that the English manufacturer is losing his dislike of publicity as to the methods used in manufacture, for there has also appeared another profusely illustrated article on Sintered Metal Bearings[36] in which the manufacture, design, application and fitting of "Reservoil" self-lubricating bearings made by the Morgan Crucible Co., Ltd. is described and illustrated.

In this short and, of necessity, incomplete survey of the trend of powder metallurgy in the last year or so, there has had to be choice of certain particular developments upon which proportionately more attention has been lavished than on the whole field of the subject. It is hoped that the notable developments in these particular lines will have conveyed the impression of real progress in this country, as well as in Europe and America. It has been said that the British always win the last battle. In the field of powder metallurgy we have won many early battles and, although we may not publish great volumes of research and development reports, we manage to keep up a steady advance, and year by year add valuable chapters to our achievement and experience.

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ALUMINIUM WIRE AND CABLE CO. LTD. announce the opening of a Midlands Branch Office at Great Western Buildings, 6, Livery Street, Birmingham, 3 (CEN. 5370).

The Zinc Consumer Industry, 1949-51

By B. Walters, M.A.

The National Smelting Co., Ltd.

The present severe restrictions on the use of zinc are not conducive to the development of new applications and in consequence the emphasis in this review is on methods of making the best use of the metal available, and thus releasing further supplies for possible future uses.

AT the present time, the world is facing an acute shortage of many materials, one in shortest supply being zinc. Following the author's review in December, 1948, supplies of non-ferrous metals continued somewhat restricted because of post-war demand, but this was followed by a recession of demand, and increased supplies of metal became available. In the spring of 1949, there appeared to be some likelihood that free trading would be resumed in non-ferrous metals, and that the London Metal Exchange would re-open. In July of that year, however, the Government made it clear that copper, lead and zinc would continue to be controlled, and at the time of writing, the Ministry of Supply is the sole supplier in Britain of copper, lead and zinc, although tin is now sold on the London Metal Exchange.

In September, 1949, came devaluation, with a sharp rise in Sterling metal prices. Early in 1950, with increasing unrest in the international situation, metal prices in the U.S.A. hardened as it became apparent that the United States Government would increase stockpiling. With the start of the Korean war, and the decision to embark on a considerable rearmament programme in the U.S.A. and in Britain, zinc for non-essential uses became very scarce, and prices rose rapidly. Prices in Britain followed closely those in U.S.A.; these changes are shown in Table I.

TABLE I.—CHANGES IN PRICE OF G.O.B. ZINC—1949, 1950 AND 1951.

Period	Lowest	Highest
	£ s. d.	£ s. d.
1949 January 1st–March 31st	106 0 0	106 0 0
April 1st–June 30th	78 0 0	106 0 0
*July 1st–September 30th	58 0 0	87 10 0
October 1st–December 31st	81 10 0	87 10 0
1950 January 1st–March 31st	85 10 0	89 10 0
April 1st–June 30th	89 10 0	127 10 0
July 1st–September 30th	127 10 0	147 10 0
October 1st–December 31st	147 10 0	151 0 0
1951 January 1st–March 31st	151 0 0	151 0 0
April 1st–June 30th	151 0 0	160 0 0
July 1st–September 30th	160 0 0	190 0 0
September 30th-date	£190	

* Devaluation occurred in this period.

Domestic production supplies about one-third of consumption, so that this country is dependent to a considerable extent on imported zinc. Because of world shortage, the Government was unable to purchase all the zinc it required, and in October, 1950, a rationing system was introduced, the allocation being based on 90% of usage during 1949. This was a greater cut than it appeared to be, because the period in question was one of reduced demand. The actual cut was equivalent to a reduction of consumption based on the first nine months of 1950 of 21% in the case of the brass and galvanising industries, and 24% in the case of the die-casting industry.

Secondary Zinc and Residuals

One immediate result of the shortage and high price of zinc was a corresponding shortage of scrap. Control of scrap had been lifted in November, 1949, so that movement within the industry was relatively free. It was inevitable, therefore, that in a number of industries, scrap was used to an increasing extent instead of virgin zinc. The price of certain grades of scrap approached, or even sometimes exceeded, that of virgin zinc. Although this state of affairs is understandable, and perhaps inevitable, it creates difficulties for two reasons.

The production of high-quality metal fabricated products depends on the availability of ingot metal of known and controlled assay, with the minimum amount of unwanted impurities. This is achieved by a steady intake of virgin metal, with a corresponding bleed out of the industry of contaminated metal in the form of scrap, where it is used for purposes where high quality is not essential, or where a particular industry is equipped with adequate purification methods.

Industries have for many years existed to convert these residuals into useful products, and reduction in supplies has meant that these industries must curtail production, with consequent similar effects on industries consuming their products, a cumulative effect reacting on the economy of any country experiencing shortage of a vital commodity.

Galvanisers, as mentioned later in this review, have endeavoured to limit dross formation, while, in addition restrictions in the use of zinc have resulted in smaller supplies of this residual being available for zinc oxide and zinc dust production. The direct recovery of the metallic content of galvanisers ashes has received considerable attention, but some difficulties have attended the use of metal thus recovered because of the high concentration of addition agents used in the bath. Chemical industries using residuals for zinc salt production have also suffered by this curtailment of raw materials.

The effect on the die-casting industry is potentially more serious. It is now well known that zinc alloy die castings are sensitive to small amounts of certain impurities, whereas other impurities although less detrimental, are nevertheless dangerous when present in relatively large amounts.

Thus, these die castings should, whenever possible, be made from virgin alloy to B.S. 1004 plus process scrap. There are, however, available quantities of scrap castings from scrapped motor vehicles and other sources. The proper place for these is the zinc dust industry, and *not* in the die-caster's pot, where their uncertain composition, dirt and oxide, plated and painted coatings, may cause the production of castings of poor quality. The inferior quality of such castings may, unfortunately, not be apparent for months or perhaps years after they leave the die-casting shop.

TABLE II.—CONSUMPTION OF ZINC IN U.K.—1945–1951, MONTHLY AVERAGE—IN THOUSAND LONG TONS.

	Total	Virgin	Secondary	Secondary/ Virgin %
1945..	20·9	14·4	6·4	44
1946..	24·1	18·0	6·1	34
1947..	26·4	18·6	7·8	42
1948..	25·9	18·6	7·3	39
1949..	23·8	16·6	7·3	44
1950..	27·3	19·7	7·6	38
1951 (Jan./June) ..	23·7	15·2	8·5	56

The brass industry was for many years the heaviest consumer of zinc. In 1949, the lead was taken for the first time by galvanising, but 1951 allocations have once again brought brass into first place. This industry has also been active in recovering as much as possible of its residuals for re-use.

Following the pattern of previous reviews, there are given the annual consumptions of zinc (Table II) and the percentage distribution among the main trades (Table III). The changes outlined above as taking place in 1951 are clearly shown in these tables. A significant feature is the high ratio of secondary to virgin zinc in 1951.

Thus, the situation in 1949, which was favourable to the development of new uses, has now changed to one of severe restriction, in which the emphasis is on making the best use of the available zinc. This is perhaps a good thing, since it is generally acknowledged that the world has in the past been too prodigal with its wealth of non-ferrous metals, but it takes a little time for industry to adjust itself to the changed situation. In these circumstances, the task of the writer who would review progress is not at first sight easy, because new uses have been stifled by shortage. However, methods of making the best use of available zinc are perhaps equally important, and will release further supplies for possible future uses.

TABLE III.—PERCENTAGE DISTRIBUTION OF ZINC USAGE IN U.K. 1948–1951.

	Brass	Galv. anising	Rolled Zinc	Zinc Oxide	Zinc Alloy Die/C.	Zinc Dust	Misc.
1948	31·2	26·7	8·9	17·4	9·3	2·3	4·2
1949	29·4	31·1	8·8	14·7	9·3	2·5	4·2
1950	30·0	30·5	8·4	14·5	10·3	2·5	4·0
1951 (Jan./June) ..	38·5	22·5	8·1	12·3	10·5	3·5	4·6

Courtesy of Fisher & Ludlow Ltd.

Fig. 1.—Motor-car rear axle casing in $\frac{3}{16}$ in. mild steel, made with Kayem 2 press tools.

Before leaving the economic situation, an excellent summary of the world zinc situation will be found in the address to the American Zinc Institute by R. L. Wilcox, Chief Non-Ferrous Metals Branch, E.C.A., "The Current World Zinc Situation." Also it is hoped that the recent meetings of the International Materials Committee will result in increased allocations to the various sections of the zinc industry in this country.

Zinc Alloys

The only new development on a commercial scale is the introduction by Imperial Smelting Corporation, Ltd. of "Kayem 2." The older "K.M. alloy," now restyled "Kayem" has been, and is still, together with its American equivalent "Kirksite," widely used as drop hammer and forming dies for aluminium and other alloys in the aircraft industry.

After the war, it became apparent that for long production runs on steel pressings as used in the motor car, refrigerator and other industries, Kayem possessed inadequate wear resistance, and a harder version, Kayem 2, was developed and marketed, the new alloy having a Brinell hardness of 145–150, as opposed to the Brinell hardness of 100 of Kayem. Press tools in Kayem 2 have proved capable of long production runs when pressing mild steel. The additional hardness has been achieved with some sacrifice of ductility, and the alloy is not suitable for drop hammer dies, for which Kayem is perfectly adequate.

A useful property of Kayem 2 is its ability to make deep pressings in heavy gauge mild steel up to $\frac{1}{4}$ in. thick. As far as is known, no long production runs have been attempted and indeed, a long tool life under these conditions would not be expected. However, it is clear that there is an obvious advantage in the use of Kayem 2 tools for the rapid production of prototype pressings in heavy gauge mild steel (Fig 1).

Kayem tools have for some time been used for making pressings for aircraft engines in austenitic stainless steel. Although mentioned in the manufacturer's brochure, it appears not to be fully realised that small amounts of zinc picked up by the steel from the tool will cause intercrystalline corrosion if the steel is heated, so that engine exhaust pressings in stainless steel made in this way must be pickled before use.[1]

Reference continues to be made to the new American alloy "Zncube" developed as a substitute for brass.[2] This is primarily a fabricating alloy; it seems probable that commitments in other directions and changed metal price relations have prevented the widespread use of this alloy. Patents[3] have also been taken out on the addition of beryllium to the accepted die-casting alloys containing zinc and aluminium, but there is at present no record of commercial use of these alloys.

Die Casting

In the die-casting industry, probably the most important event is the visit, sponsored by the Anglo-American Council on Productivity, by die casters from this country to America. The team has recently returned, and their official report is awaited.

The production of very small die castings in specialised machines has recently become a feature of interest, and a number of these are shown in Fig. 2.

The passage of time has permitted the collection of data on the long term ageing at room temperatures of zinc alloy die castings to B.S. 1004; results are not yet

published, but it will shortly be possible to dispense with accelerated dry ageing tests at 95° C. as an indication of the behaviour of die castings after normal ageing, and to substitute the actual values.

One point can now be stressed; unpublished work has served to underline the importance of the small amount of magnesium in die castings to B.S. 1004 in preventing intercrystalline corrosion arising from small amounts of residual impurities in the alloy. In this connection, it is indeed unfortunate that the shortage of zinc alloy increases the danger of excessive scrap use and remelting. This, particularly if carried out in the presence of fluxes, inevitably reduces the magnesium content by selective oxidation or flux reaction.

Galvanising

Of the various grades of zinc, that in shortest supply has been G.O.B. As the galvanising industry depends on this grade of zinc, the cut to this industry was rather more than the 21% mentioned above. While the shortage began to be felt in October, 1950, the rationing scheme did not come into operation until January, 1951, but when it did come, general galvanisers received only around 50% of their 1950 purchases. It has been possible for galvanisers to carry on by using stocks, but this has an obvious limit, and the future is by no means bright.

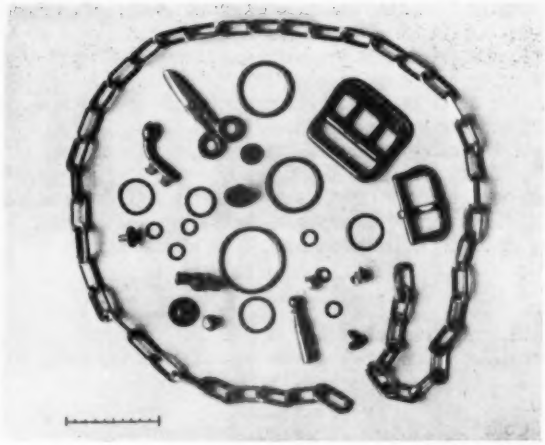
It is, however, in the galvanising industry that the most technological progress can be reported during the last two or three years. Galvanising is a long established industry. With zinc cheap and in plentiful supply, it did not matter if heavy coatings were put on the work, and if a fair amount of zinc disappeared in secondary products, dross, the zinc-iron layer which sinks to the bottom of the bath, and in ash and flux skimmings from the surface of the bath. These found a ready market at a fair price, and were easily replaced by virgin zinc. Much attention has recently been paid to uniformity of coating thickness, and to methods of minimising dross production, a great deal of this work having been carried out by the British Iron and Steel Research Association and British Non-Ferrous Metals Research Association.

Two important events have also taken place: the First International Hot Dip Galvanising Conference held at Copenhagen in July, 1950, and the visit of a team to America under the auspices of the Anglo-American Council on Productivity, reports of both of which have been published.[4, 5]

International Conference

The Copenhagen conference was organised by the Zinc Development Association on behalf of the Hot Dip Galvanisers Association, a body recently formed along lines similar to those of the Zinc Alloy Die Casters Association. The papers presented at this conference, and the discussions which followed, give an up-to-date picture of the progress made in the industry during recent years. The report is worthy of the most careful study, and it is only possible here to touch on some of the most salient aspects.

Production of Residuals. Systematic examination of a number of industrial plants has shown that production of zinc ash, which is mainly metallic zinc with entrained oxide, can vary from around 1–30%, the governing factor being the extent of the flux cover, care in skimming, and control in speed of immersion and withdrawal of the work.



Courtesy of George Goodman Ltd.

Fig. 2.—A number of very small Mazak die castings—reference line is 1 in. long.

Production of dross can vary between 9% and 38%. There is now ample evidence to prove the importance of washing to remove iron salts after pickling. Iron salts carried into the bath are reduced by molten zinc to metallic iron, forming zinc-iron compound or dross. The acid used for pickling has an indirect bearing on dross formation because, in the case of hydrochloric acid, iron chloride acts as a fluxing agent, and in this respect is beneficial. On the other hand, if sulphuric acid is used, iron sulphate has no fluxing action. It is also interesting to speculate whether, as chlorides are in general more reactive than sulphates, less dross would be produced due to imperfect washing if sulphuric acid pickle were used: so far there appears to be no evidence either way. Dross formation is also reduced by careful bath temperature control, and by optimum rate of throughput.

Bath Additions. Much light has been thrown on the influence of the various bath additions, particularly aluminium. Hughes[6] has shown clearly the effect of small additions of this element in reducing the thickness of the alloy layer, and improving the ductility of the coating. Fagg[7] has confirmed this, but has shown that with increased aluminium contents—i.e., over 1%, the thickness of the alloy layer increases rapidly, particularly on prolonged immersion.

Because of the attack of aluminium on the conventional chloride fluxes, 0·05% is said to be the absolute maximum which can be employed when using these fluxes, even by the dry galvanising process, in which work is pre-fluxed and dried. In this connection it may be mentioned that much research has been devoted to fluxes based on other salts such as fluorides, and containing aluminium, in the hope of finding a flux compatible with aluminium, but it is doubtful whether any real success has been achieved, and they have certainly not been adopted to any extent in this country.

The effect of small amounts of aluminium on the corrosion resistance of the zinc coating is slight and probably beneficial. Hughes[8] has referred to the lowered corrosion resistance when both aluminium and lead are present together: lead is normally present up to about 1%, because G.O.B. or Prime Western is the favoured grade of zinc for galvanising. This observation is in line with the severe effect of lead in lowering the corrosion

resistance of zinc alloy die castings, this effect being dependent on the presence of aluminium: tin has an effect similar to lead. With the present very small amounts of aluminium present in the coating, the lowering of corrosion resistance is small. The present author considers that should methods be evolved of introducing larger amounts of aluminium, the possible lowering of corrosion resistance if lead or tin are also present may become significant.

The above are only a few of the factors discussed at Copenhagen, but the report is a contribution of permanent value to the literature on hot dip galvanising.

Productivity Report

The Productivity Report on hot dip galvanising practice in the U.S.A. merits careful reading, and again it is only possible to pick out a few technical points for comment.

Compared with galvanising in this country, the larger amount of work on products of a similar type permit more specialist galvanising, and a far greater degree of mechanical handling of work through the pickling and galvanising baths is possible. This, in turn, allows a high degree of control, and a uniform product. There is a noticeable tendency for components to be designed to facilitate the galvanising operation, for instance in the case of hot water cylinders. As in Britain, Prime Western zinc is used exclusively for galvanising.

A fairly recent development in the U.S.A. is the Wean process, using granulated zinc for galvanising small parts such as nails by tumbling in a heated barrel. Advantages accrue from mechanisation from start to finish and very small production of residuals. This process is also used in this country.

Also of interest is the electrical low-frequency induction heating of baths by induction units placed on the sides of the bath.[9] The baths can be, and are, refractory lined, and a lining life of up to 10 years is expected. The agitation which occurs greatly assists in maintaining temperature and composition uniformity, and the location of the inductor units near the top of the bath is said not to prevent the settlement of dross, but this point still appears to be open to question. Inductors up to 333 kW are available, and a number may be used in a single bath. This method of heating is in use at a few plants, and as in the case of all electrical heating methods, temperature control, a most important factor in galvanising, is easy.

It is now quite clear that the life of all zinc coatings is a direct function of their thickness. It is to be hoped, therefore, that, while energy is being directed to saving zinc, this important fact will be kept to the fore.

Miscellaneous

Corrosion. Lodder[10] has reported that chromate treatment goes very far in preventing attack on zinc alloy die castings to B.S. 1004 by wall plasters containing chlorides, and that virtually complete protection is given by following this with suitable paint coatings. This has particular application to electrical conduit fittings and switch gear. Another problem which continues to receive attention is that of the reversed polarity of zinc and iron in water systems, which results in rapid attack of the iron. The occurrence of white rust on zinc in conditions of damp storage with limited air circulation has also been investigated, by Gilbert and Hadden:[11] while the mode of occurrence is still not clear, its mitigation by chromate treatment is confirmed.

Rolled Zinc. The dry battery industry continues to absorb a large proportion of zinc rolled in this country, the popularity of zinc roofing shown on the Continent not being evident here. On the metallurgical side a paper by Roberts and Walters[12] has reviewed in some detail production methods, grades of zinc and types of alloys used, and metallurgical problems connected with rolling.

Zinc Pigments. Extending his work on cementiferous paints, Mayne[13] finds that zinc dust paints based on di-potassium and tri-potassium phosphates are superior to the earlier cementiferous paints based on chlorides. The paints are cheap and easy to prepare, and have appreciable storage life. The use of zinc tetroxychromate is increasing, particularly as a constituent of etching or wash primers based on phosphoric acid and used on light alloys.

Finishing. The nickel shortage is affecting the plating of zinc alloy die castings, but it is possible that the new tin-nickel alloy[14] may be a temporary substitute, provided precautions are taken to prevent diffusion of tin into the zinc alloy.

Acknowledgment

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Productivity Reports

Two more Productivity Reports have appeared recently, dealing with "Education for Management" and "Metal Finishing." Copies are obtainable from the Anglo-American Council on Productivity, 21, Tothill Street, London, S.W.1. The prices, post free in each case, are 4s. 6d. and 3s. 6d., respectively.

THE Technical and Scientific Register of the Ministry of Labour and National Service has moved from York House, Kingsway, London, W.C.2., to Almack House, 26/28, King Street, St. James's Square, London: S.W.1. Telephone No. TRAfalgar 7020.

Magnesium and Its Alloys in 1951

By R. G. Wilkinson, B.Sc.

Deputy Chief Metallurgist, Magnesium Elektron, Ltd.

The stocks of pure magnesium and primary and secondary grades of magnesium alloys remaining after the war have now been exhausted and production plants are being re-opened. The rearmament programme will, no doubt, result in increased consumption and, in this article, reference is made to recently developed alloys with improved mechanical properties which will find increasing application in many spheres.

THIS brief review of the present status of magnesium and its alloys is written during a period of shortage of the metal consequent upon nearly complete exhaustion of stocks coupled with increased demands for rearmament purposes, and in June last consumption of all but secondary alloys was placed under Government control. Following the war, this country and, more especially, the U.S.A. were left with large stockpiles of both pure magnesium and primary and secondary grades of the standard alloys containing aluminium and zinc, and all English production plants and nearly all in the U.S.A. had been closed down by the end of 1947. From that time civilian usage, together with the remaining military applications, consumed the magnesium stocks at an ever increasing rate, resulting in the shortage which became acute in the U.K. early in 1951.

Courtesy of the Standard Motor Co. Ltd. and Sterling Metals Ltd.

Fig. 1.—Castings in Elektron "C" alloy for the Ferguson tractor, total unmachined weight 63¼ lb.

The main British use of magnesium alloys has been in the form of castings, representing about 75% of the average annual output. The bulk of this tonnage has consisted of secondary material, namely Elektron "Commercial" alloy, which is similar to the old-established D.T.D. (now B.S.-L) alloy Elektron A8, except that its compositional limits are wider. Castings in this material have been for some years competitive in price with corresponding aluminium alloy castings, and the weight saving, together with a notable advantage in machinability, have resulted in wide usage. Two of the most important applications are illustrated in Figs. 1 and 2. Sterling Metals, Ltd., have now produced well over 200,000 sets of the Ferguson tractor castings, the peak output of their mechanised foundry at Nuneaton being 1,600 sets per week. The facts that very large numbers of these tractors have been exported to most parts of the world for some years, and that the highly stressed Elektron unit has proved entirely satisfactory, provide ample justification for the use of magnesium. It is especially worthy of note that, despite the exposed position of the transmission unit and the arduous service conditions in a variety of climates, no corrosion troubles have been reported. Warp beams and flanges have proved another particularly successful application, and there must now be scores of thousands of such castings in use in the textile mills.

Production of wrought alloys in the U.K. has been comparatively small and civilian applications in this field few. But it is worth noting that large numbers of the warp beams have been manufactured from extruded Elektron tube. In the U.S.A., however, there has been a striking trend towards the increased use of wrought magnesium alloys, particularly in the form of extrusions,

TABLE I.—TENSILE TESTS OF SPECIMENS TAKEN FROM ALL REPRESENTATIVE PARTS OF Z5Z MAIN LEG CASTING FOR LARGE UNDERCARRIAGE.
(This casting, unmachined weight 360 lb., is illustrated in Fig. 3).

Bar No.	Section	0·1% P.S. tons/sq.in.	U.T.S. tons/sq.in.	Elongation %
1	1½ in. Unchilled	8·2	14·6	5·0
2	1½ in. Chilled	6·4	11·0	2·8
3	1 in. Chilled	8·0	13·0	3·4
4	1 in. Chilled	8·0	13·2	4·0
5	1 in. Chilled	8·0	14·2	5·2
6	1½ in. Unchilled	8·0	13·2	3·8
7	1½ in. Unchilled	8·6	14·8	5·0
8	1½ in. Chilled	7·6	10·6	1·0
9	1 in. Chilled	7·6	13·0	2·4
10	1 in. Chilled	8·0	14·6	5·6
11	1 in. Chilled	8·4	14·8	5·0
12	1½ in. Chilled	8·0	14·2	5·0
13	1 in. Unchilled	8·0	13·8	3·4
14	3 in. × 4 in. Unchilled..	7·9	14·0	4·2
15	3 in. × 4 in. Unchilled..	—	14·5	4·0
16	1½ in. Chilled	7·8	13·4	3·2
17	1½ in. × 4 in. Chilled ..	8·0	15·6	7·0
	SPECIFICATION MINIMA IN D.T.D.721.. ..	8·5	15·0	5·0

and, in extreme contrast to the position in this country, the total tonnage of wrought products shows signs of exceeding that of castings.

Following the depletion of stocks, and coinciding with the rearmament drive, at least six production plants have now been, or are being, reopened in the U.S.A., two are in production in Canada, and in England the Clifton Junction plant of M.E.L. was brought into partial operation in July of this year. On the Continent of Europe small plants are working in France and Italy, and a large one in Norway is being brought into use.

Considerable quantities of pure magnesium have been imported into the U.K. from America and Canada for some time, and unless further production capacity is made available here such purchases seem likely to continue.

Casting Alloys

In an article in the December, 1949, issue of *Metallurgia*, the writer described the zirconium-containing alloys Elektron Z5Z (magnesium–0·6% zirconium–4·7% zinc), ZRE 1 (magnesium–0·6% zirconium–2·5% rare earth metals–2·5% zinc) and MCZ (magnesium–0·6% zirconium–3% rare earth metals), then about three years old in foundry experience. During the last two years, the great promise shown by these materials has been fully confirmed and their production has steadily increased.

One of the principal advantages of the high proof stress structural alloy Z5Z is that it shows considerably greater uniformity of tensile properties with varying section of the casting than is the case with the old magnesium – aluminium – zinc – manganese alloys, together with a much closer approach to separately cast test bar values. These facts were of course known when the alloy was first introduced and have materially contributed to its growing popularity with aircraft designers. Z5Z has recently been specified for a very large Dowty undercarriage main leg casting (Fig. 3), of unmachined weight 360 lb., considerably larger and heavier than the Messier undercarriage of the Handley Page Halifax bomber, some thousands of which were cast in Elektron A8 during the war. In foundry control tests, 17 tensile specimens were taken from this new Z5Z casting, covering chilled and unchilled sections varying from 1 in. to 3 in. in thickness, and from all representative parts of the casting, and the results (Table I) were really remarkable in their consistency, and in comparison with D.T.D.721 minima. It may, perhaps, be mentioned that the inclusion of a mandatory minimum proof stress in D.T.D.721 represents an important innovation in the history of light alloys, whether of aluminium or magnesium base.

Most of Britain's new aircraft have been taking-off and landing on Z5Z alloy wheels for the last three years, and their performance, together with the results of special laboratory tests carried out by the Dunlop Rim and Wheel Company, would appear to indicate a successful future for such a type of alloy in this application. It is probable that the zirconium-containing alloy is proving superior to A8 because it is sounder and more free from stress raisers liable to promote fatigue failure In fact, many sound wheels have been produced in Z5Z in cases where the complexity of design made their casting to an acceptable standard of quality in A8 impracticable.

An interesting variant of Z5Z alloy has just been

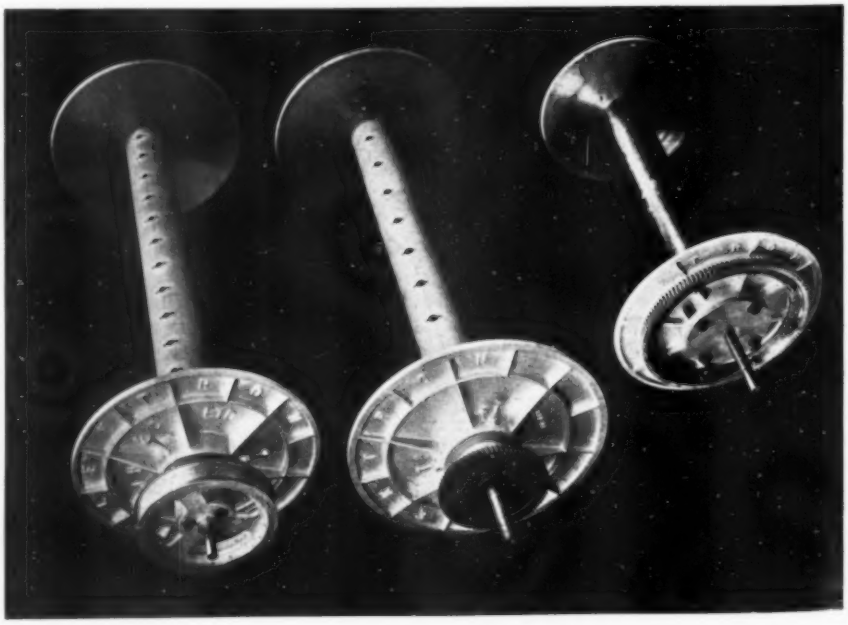


Fig. 2.—Weavers' beams made entirely of Elektron except for axles, bolts and, in the case of the centre flange, the patent let-off gear. Each weighs less than half of the old type beam it replaces.

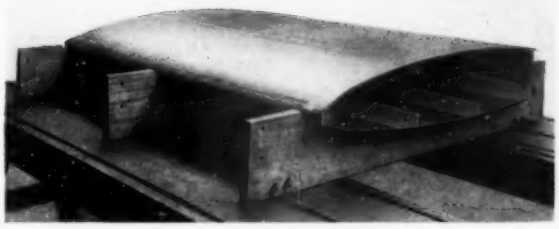
introduced. This material, termed RZ5, contains 1·25% rare earth metals in addition to the normal zinc and zirconium alloying elements. This variation in composition involves some sacrifice in ultimate stress and elongation, but the proof stress is reduced only slightly (a mandatory minimum of 8·0 tons/sq.in. is proposed for the draft D.T.D. specification against the 8·5 tons/sq. in. for Z5Z in D.T.D.721). On the credit side, however, are the facts that the addition of rare earth metals results in an even higher degree of freedom from microporosity, leading to pressure tightness; it conduces to argonarc weldability of the alloy; and it makes possible the production of certain special "spidery" castings which are difficult to make in straight Z5Z without the occurrence of hot cracking (Z5Z castings cannot be repaired by argonarc welding because of weld junction cracking).

Courtesy of Dowty Equipment Ltd. and Sterling Metals Ltd.

Fig. 3.—Main leg casting in heat treated Z5Z to D.T.D.721 for large undercarriage. The weight of this casting is 360 lb. unmachined.

There is also a recent development in connection with the ZRE 1 and MCZ types of creep-resistant alloy. These two materials have virtually the same normal and elevated temperature tensile properties and creep resistance up to 250° C. (equivalent in creep to the aluminium base alloy RR50). The addition of zinc in the case of ZRE 1, however, was made in the interests of castability. A third alloy of this type is now being introduced for marine application, e.g. jet and propeller turbine engine components for deck landing aircraft. Named ZRE 0 and containing only 0·5% zinc in addition to zirconium and rare earth metals, this new material has unchanged tensile and creep properties, but is designed for optimum corrosion resistance under severe service conditions.

When ZRE 1 and MCZ were introduced to the aircraft engine industry, they were recommended as creep-resistant alloys at temperatures up to 200° C., or at



Courtesy of the Bristol Aeroplane Co. Ltd.

Fig. 4.—Test section of wing, length 6 ft., fabricated in an Elektron magnesium-zirconium-zinc alloy and incorporating both span-wise and chord-wise argonarc welds made on an automatic machine similar to that shown in Fig. 5.

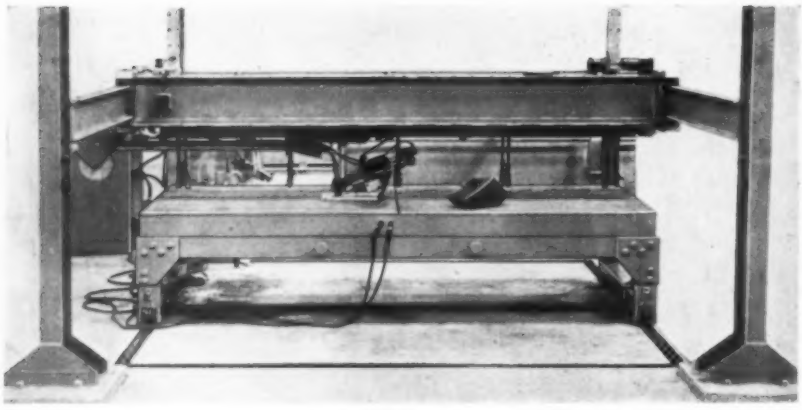
highest 250° C., and in the main they have been put into service within this temperature range, although there are cases on record of their successful use at higher temperatures. In the Metallurgical Research Department of M.E.L., intensive work over the last two years has been directed towards the discovery of alloys which would in all circumstances be creep resistant up to 350° C., to meet the engine designers' demands involved in the development of jet and propeller turbine engines of increasing power. It is not permissible at present to disclose the successful alloy composition, but it can be said that the research at Clifton Junction has led to the formulation of two alloys based upon the magnesium–zirconium system with quite remarkable properties. Alloy "A" has the normal temperature tensile properties of ZRE 1, but shows satisfactory creep resistance up to 350° C. instead of merely 250° C., and alloy "B" has the normal temperature tensile properties of Z5Z (high proof stress) with a creep resistance up to 250° C. equivalent to that of ZRE 1. A foundry trial of alloy "A" has already been carried out and both tensile and creep tests have been run on the casting produced. The creep values confirmed those previously obtained on laboratory cast test bars, and the tensile tests disclosed a remarkable characteristic of the alloy in yielding higher figures in the thicker unchilled sections than in the thinner chilled areas.

Further development of these alloys is being intensively pursued, and there seems no doubt that they will find important rôles in future prototype engines.

Wrought Alloys

In contrast to the casting field, usage of wrought magnesium alloys in this country since the war has been disappointingly small and, as previously mentioned, the U.S.A. has taken a most definite lead in this respect. The situation is the more disappointing since Elektron ZW 3 (magnesium–0·7% zirconium–3·0% zinc), having clearly superior properties to the older alloys AM503, AZM and AZ855, has been available against D.T.D. specifications in all wrought forms for more than five years. The corresponding American alloy, Dowmetal ZK60A (magnesium–0·6% zirconium–5·7% zinc), is used extensively in extruded form, especially for the floor beams of transport aircraft.

Despite this lack of encouragement in the U.K., however, production and fabrication development have proceeded steadily, and this policy is being justified by serious interest in the aircraft industry in the possibility of using the alloy for primary wing and fuselage structures of monocoque or "lobster claw" construction.



Courtesy of the Bristol Aeroplane Co. Ltd. and Birmetals Ltd.

Fig. 5 (left) Automatic argonarc welding machine.

Courtesy o British Overseas Airways Corporation

Fig. 6.(right)An all argonarc welded experimental aircraft chair frame constructed by M.E.L. in Elektron magnesium-zirconium-zinc alloy tube.

This potential application is of course based upon the advantage of utilising the lightness of the material by the adoption of thick and correspondingly robust skins capable of maintaining accurate aerodynamic form, whilst achieving very substantial economies in man-power through elimination, or great reduction in the number, of internal stiffening elements. Valuable fabrication development work has been carried out on this possibility, in the main based upon the extensive use of automatic argonarc welding, and procedures are being successfully evolved for the production of wings (Fig. 4). Argonarc welding machines (Fig. 5) have been developed, and tests have shown weld strength efficiencies up to about 80% to be achievable.

Experience over the last few years has shown that, although ZW 3 can be argonarc welded satisfactorily by the automatic process, manual welding of joints involving substantial constraint is impracticable because of the risk of weld junction cracking. To meet this difficulty, a variant of ZW 3, ZW 1 with the zinc content reduced to 1·5% has been introduced, and this alloy is entirely free from weld cracking tendencies under all conditions. The reduction in zinc content involves only a small sacrifice in mechanical properties—of the order of 1–2 tons/sq.in. Fig. 6 shows an experimental aircraft chair fabricated by argonarc welding an alloy similar to ZW 1, but in this case containing 2·0 instead of 1·5% zinc.

The advantages of the ZW-type wrought alloys lie in their excellent hot workability from cast stock, in their superior tensile, compressive and toughness properties, in their ease of fabrication, and in their freedom from stress corrosion tendencies in service. They have now superseded the older alloys in many secondary applications in aircraft, and in some other fields.

Cracking Control in High-Tensile Steel Welding

SEVERAL advantages are to be gained from the use of low alloy steel in the building of bridges and similar structures. The increased tensile strength of such steel results in a considerable saving in weight of the integral parts of the structure, with a consequent economy in transport and erection.

The welding of the higher tensile steels, however, is accompanied by a type of failure which is known as hard zone cracking. The problem of welding these steels satisfactorily, without cracking, is one which interested the Ministry of Supply. Accordingly, the British Welding Research Association undertook an investigation into this problem, the work falling under the guidance of the F.M.8 Committee. In the August issue of *Welding Research*, the journal of the Association, a report of this work was presented under the title of "Control of Cracking in the Metal Arc Welding of High Tensile Structural Steels."

A typical structural unit was fabricated from a selected steel, and cooling rates measured in the heat affected zone adjacent to each weld. All the welds were subsequently sectioned and examined microscopically for cracks. In this way cooling rate was related to the occurrence and extent of cracking. It was found that a critical cooling rate existed for each type of electrode when used to weld the given steel. When this critical cooling rate was exceeded, hard zone cracking occurred.

This paper, which was first presented to the International Welding Congress in July, describes the investigation and the results obtained. The results are used to show how cracking can be avoided by making welds of a minimum fillet size without the use of preheat.

Mention is also made of a new weldability test which can be made on plates of various thicknesses to give different grades of severity. This test, called the Controlled Thermal Severity (C.T.S.) test, can be used to assess the weldability of a steel, to determine the suitability of an electrode, or to simulate conditions in a given structural joint.

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Copper and Copper Alloys
A Survey of Technical Progress during 1951

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Continued progress has been made during the past year in the metallurgy of copper and its alloys, and in the various sections of this review the author discusses the principal items of interest concerning such subjects as raw material resources, extraction, fabrication, finishing and properties.

IN common with most other industries, that concerned with copper and its alloys has felt the impress of rearmament and the economic disturbances which inevitably accompany it. A year or two ago it seemed that the industry was well on the way to recovery from the aftermath of war: deliveries were achieving normality and supplies seemed adequate to meet current demands. Within the last twelve months, however, the picture has entirely changed. Once again, owing to the paramount needs of defence, certain restrictions have been imposed, and shortages, with their attendant protracted deliveries, have already to be faced.

Troublesome though they be, however, such shortages need not be regarded with undue alarm. They are primarily a manifestation of the present international tension and do not arise from any immediate deficiency in the available reserves of copper ore. This fact was made abundantly clear during a recent discussion on metal economics held by the Institute of Metals, where it was stated that sufficient unworked copper ore to produce approximately 100 million tons of copper has probably already been located. At the present rate of production this would be sufficient to supply the requirements of industry for about 44 years. This, moreover, represents by no means the whole reserves even of existing mines, and it was pointed out that many mines which reported limited reserves during the early part of the century are still in operation, the reserves currently quoted being in many cases larger than they were before. It should be added that prospecting is continually in hand.

In spite of the generally less optimistic commercial outlook, a fairly normal year of technical progress and development has been reflected in the numerous publications which have made their appearance on matters connected with copper and copper alloys. The purpose of this review is to outline a few selected examples from among them.

The Production of Copper

Prospecting and Mining. Although prospecting for copper is being actively pursued all over the world, recent reports have given special prominence to Canada and the southern parts of the African continent. Near Bathurst Inlet, Northern Canada, where copper has long been known to occur, a new prospect is reported,[1] while a deposit discovered as early as 1928 on the Rankin Inlet of Hudson Bay is claimed to be rich in nickel as well as containing appreciable copper.[2] Mixed ores of copper and zinc have been found in the Kreighoff region of Quebec,[3] while the reserves at Lynn Lake are reported to be considerably more extensive than has been thought up to the present.[4, 5, 6] Expectations of further finds in this region are high. Plans are in hand for the development of copper deposits near New Hazelton,[7] and, across the border, the possibilities of a very large new mine in Northern Michigan are being investigated.[8]

In the Mount Ayliff area of Pondoland, South Africa, bore-holes have revealed promising quantities of copper, nickel and cobalt,[9] while possible developments in connection with copper, nickel and platinum are reported from Southern Rhodesia.[10] Notes by Murdock[11] on the production of copper at the Lubumbashi smelter of the Union Minière du Haut Katanga include the information that the Prince Leopold Mine, from which this smelter draws its ore, has an estimated life of ten years. Mention has also been made of the existence of 1·7 million tons of ore assaying 4·2% of copper together with small amounts of nickel and cobalt in Uganda.[12]

Considerable activity is reported[13] in Jugoslavia, especially at the Bor mine, now claimed to be Europe's largest producer of copper. During 1950, some 40,000 tons of blister copper were derived from this source, while new mines are being investigated and developments in connection with refining and fabrication of the metal are in progress. A recent review of the mining and metallurgical industries of Norway[14] indicates that the production of copper there is not extensive, the chief mines being situated at Rösos, Sulitjelma and Thamshavn. On the other hand, from Finland comes the news[15] that operations are being recommenced at the Nivala copper-nickel mine, which was originally opened in 1941 but closed again in 1947, while reports from both India[16] and the Philippines[17] indicate activity in the production of copper in the Far East.

Ore Dressing and Extraction. In connection with ore dressing, Myers and Lewis,[18] of the Tennessee Copper Company claim that the development of a large slow-speed ball mill in closed circuit with a hydro-oscillator has resulted in a grinding efficiency 28% higher than that obtainable with conventional units. This may well herald a trend towards the use of larger mills working at slower speeds than are at present adopted.

In the Review for 1950, brief descriptions were given of the flash smelting of copper concentrates and of the separation of copper and nickel sulphides by flotation methods. It is interesting to note that the first of these processes has now been patented in this country,[19] while the second appears to have been developed, presumably independently, in Germany by Schlecht and Schlecht.[20]

It is stated that the flash smelting plant of the International Nickel Company of Canada, mentioned in the Review for 1950, is soon to be in operation.[21]

An able review of the design, construction and operation of modern copper reverberatory furnaces has been published by Dennis,[22] who emphasises the importance of the nature and quality of the refractories and of the technique of their use, paying particular attention to the development of the suspended roof of magnesite brick.

In view of the present shortage of sulphur, a further process for the recovery of this element from cupriferous pyrites is of interest.[23] The pyrites is first roasted to drive off some of the sulphur, which is collected, and the porous residue is then treated with sulphuric acid to form a solution of ferrous sulphate with the liberation of sulphuretted hydrogen gas. This gas is partially burned to convert a proportion of it into sulphur dioxide, and from the mixture so obtained liquid sulphur and steam can be produced catalytically. The solution of iron sulphate is electrolysed to recover the iron and regenerate the sulphuric acid, while the insoluble copper sulphide remains as a sludge from which the copper can be extracted by orthodox methods.

A paper by Larson[24] has dealt with the introduction of equipment for the mechanical punching of convertor tuyères at the McGill Smelter of the Kennecott Copper Corporation. After six years of effort, concentrated on the development of a mechanically efficient design, a pneumatic device has been evolved which appears to be both robust and effective. It is claimed that the rate of copper production is from 15–20% greater than with hand punching, using the same grade of matte.

Refining. Two new electrolytic refining projects have been reported. One of these, owned by the Kennecott Copper Corporation is situated at Garfield in Utah and has an output of 12,000 tons per month,[25] while the other is sponsored by a combination of French and Belgian interests which are to operate a refinery near Limoges.[26] Bradford[27] describes extensions to the electrolytic plant of the Rhodesia Copper Refineries, Ltd. at Nkana, while Kunkle[28] gives a detailed account of the Phelps Dodge refinery at El Paso.

While copper of exceptionally high purity has been produced by special methods of electrolytic refining, both in this country and in the United States, it is interesting to note that copper of comparable purity has now been made in Japan.[29] During the process of purification, manganese dioxide was used to occlude the impurities, presumably in the form of hydroxides, from an ammoniacal solution of copper salts.

Experiments on the refining of secondary copper alloys have been reported by Glassenberg, Mondolfo and Hesse,[30] who found the sequence of removal of the common elements from molten copper by oxidation to be: aluminium, manganese, silicon, phosphorus, iron and, lastly, zinc. The use of oxygen in place of air showed little advantage in the refining of a typical brass alloy, but there were indications that oxygen can improve the removal of iron when high concentrations of zinc are also present.

Foundry Practice

Melting Furnaces. Evans,[31] reviewing furnaces for non-ferrous melting, makes the important point that under modern conditions of high metal cost, melting losses are of more importance than fuel consumption. He states that, in Great Britain, the cost of coke per ton of metal melted is roughly equal to a melting loss of 0·5% on a copper-base alloy. Thus if the melting loss rises from 0·5–1%, then this loss costs twice as much per ton of output as the coke used. The thermal efficiencies for the various types of furnace employed in non-ferrous practice differ considerably but, with the exception of those using electricity, none approaches the efficiency of the cupola which is the normal equipment of the iron foundry. While Evans does not mention the possibilities of the cupola for melting copper-base alloys, proposals to this end have been put forward from time to time. It has recently become known that cupola melting of bronze was practised in Japan during the war, owing to the shortage there of graphite crucibles.[32] The product was said to be satisfactory, even when poor quality coke was used, but the melting losses were comparatively large.

Continuous Casting. It is believed that a continuous casting machine for the production of brass billets recently installed in the works of Messrs. James Booth & Co., Ltd., of Birmingham,[33] is the first to operate on an industrial scale in the British brass industry. It is of the Junghans-Rossi type. A patent reveals that the Junghans-Rossi process has now been applied to the production of tube,[34] a special feature of the apparatus being that the heat is abstracted through the internal mandrel which is water-cooled while the outer walls of the mould are heated. This leads to radial crystal growth. Another patent, by Goss,[35] aims at the development of a continuous casting machine which will retain perfect alignment even though it may expand freely in every direction while being subjected to the thermal shock of liquid steel or non-ferrous metals.

Though little has lately been heard of the Hazelett process for the continuous casting of sheet, a recent paper[36] states that gradual improvements have been effected during the past fifteen years. Originally, the mould pocket was formed between two cylindrical rolls, but in the later developments the arrangement consists of two water-cooled steel belts. Liquid metal is poured between these, as they pass over successive pairs of guide rolls, and emerges as a thin continuous strip of solid metal. Brass has already been successfully cast with this machine.

In the course of a discussion of continuous casting sponsored by the Birmingham Metallurgical Society, Chadwick[37] stressed the economic advantages of continuous casting in the production of copper and copper alloys, while Wood[38] dealt in considerable detail with the more theoretical aspects of the subject.

Other Casting Processes. A long series of articles on the principles of die design for die casting has been written by Barton and Barton,[39] while Eggleston and Gregory[40] have discussed the composition and properties of a number of copper-base alloys suitable for the gravity die casting process. The gravity die casting of brass is troublesome because of the accretion of a drossy mixture of zinc and zinc oxide on the die surfaces. To overcome this, Robertson and Browse[41] have patented the use of a die coating containing a metal oxide capable of being reduced to a lower oxide by zinc at the pouring temperature; for example, an aqueous suspension of manganese dioxide mixed with an alcoholic solution of tannic acid.

The "Croning" process, in which a thin shell of resin-bonded refractory is coated on the pattern, has been

described in a previous Review. Originally developed in Germany, it is now being exploited to a very considerable extent in the United States, as mentioned in the report of the Productivity Team on bronze and brass founding.[42] An article by Czygan[43] describes how high-speed machinery has been applied to the process which, it is claimed, may well revolutionise foundry practice, especially in cases where castings are required in very large numbers.

The Advantages of Leaded Gunmetals. From the report of the Productivity Team just mentioned[42] it appears that the well-known "85 : 5 : 5 : 5" leaded gunmetal (B.S. 1400–LG2) is used in the United States for many applications for which bronzes or gunmetals richer in tin would be favoured on this side of the Atlantic. This clearly makes for simplification of standardisation and efficiency in the control of scrap and organisation of metal stores. In the light of this information, Mantle[44] has described earlier experimental work by the British Non-Ferrous Metals Research Association showing that leaded gunmetal of the composition in question has marked advantages in consistency of quality and pressure tightness over several other bronzes and gunmetals, even when cast under unfavourable conditions from relatively gassy melts. He urges that this excellent alloy should be adopted as widely as possible.

Castings in Some Special Alloys. For pressure-tight castings, Daase[45] recommends the use of a brass containing silicon because of its high fluidity when molten, while Vanick[46] suggests an alloy containing copper 85%, nickel 2·5–3·5%, tin at least 2·5%, lead and zinc each 4–6%, for the same purpose. The same author, in other papers,[47, 48] has described the properties and applications of the heat-treatable nickel bronzes with special reference to the production of gears and bearings. He points out that lead, beneficial to machinability, should be kept to a minimum where maximum strength and toughness are desired, but that it is a valuable addition for bearing applications.

In the production of cast copper-lead bearings, a small amount of sulphur is frequently added to improve the distribution of the lead. Ruhenbeck[49] states that the optimum ratio of lead to sulphur is approximately 100 to 1, and adds that the presence of iron somewhat in excess of its limit of solid solubility in copper gives rise to a fine grained spherical type of structure, which is associated with improved strength and bearing qualities. The same author[50] also reviews methods of obtaining an optimum bond between the bearing metal and the backing in the production of steel-backed copper-lead bearings.

While beryllium copper now ranks as a well-known material in wrought forms, it is less widely recognised that it also has important applications in the form of castings. Among these, non-sparking tools such as hammers, chisels, wrenches and pliers deserve special mention, while Richards[51] has described the application of this interesting alloy to the production of small components with accurate dimensions by the investment casting technique : included in the paper are some useful instructions for colouring beryllium copper. Hildebrandt[52] has studied the tensile properties of beryllium copper alloys containing from 1·78–2·82% of beryllium when cast in moulds of baked core sand. After heat treatment, tensile strengths of between 50 and 70 tons/sq. in. were obtained, with hardness values up to 420 diamond pyramid number.

The Repair of Castings. The aim of every reputable foundry is to produce castings which are as nearly perfect as possible, but even when every precaution is taken defects occasionally arise which can be repaired or otherwise corrected. A paper[53] presented to the Institute of British Foundrymen gives a comprehensive review of the available methods of reclamation for both copper and aluminium-base alloy castings by such expedients as burning, welding, brazing, soldering, annealing, impregnation, caulking, plugging, plating and spraying, with recommended practices in each case and the types of service for which the repaired castings are suitable.

The Mode of Solidification of Castings. A detailed study has been made by Ruddle and Mincher[54] of the effect of constitution on the manner in which an alloy solidifies. Based on these researches, the authors have been able to calculate the periods required for the solidification of castings under given conditions, and this may well prove to be of practical value in planning production schedules in the foundry.

Fabrication

Fundamental Aspects of Deformation. The Spring Symposium of the Institute of Metals was this year devoted to the cold working of non-ferrous metals and alloys. The first paper in the series of five[55] reviews the mechanism of deformation from the fundamental standpoint. For polycrystalline copper and alpha copper alloys, deformation during the earlier stages takes place mainly by slip on those crystallographic planes which are most densely packed with atoms. Under severe cold-working processes, however, deformation occurs by shear on planes which are determined more by the direction of the applied stresses than by the orientation of the individual crystals, except in so far as the progressive development of preferred orientation in the worked material causes the orientation of the crystals themselves to be dependent upon the direction of the applied stresses. Preferred orientation in the cold-worked material generally leads to preferred orientation after annealing, with attendant directional properties such as those which give rise to the formation of "ears" around the periphery of deep drawn cups.

Rolling. In his contribution on the cold rolling of non-ferrous metals to the Symposium, Davies[56] contrasts the American practice of cold breaking down, even in the largest and most modern mills, with the normal British technique of hot rolling such materials as the *alpha* brasses. Most of the American *alpha* brasses contain sufficient lead, either as an intentional addition or as an impurity, to make them unsuitable for hot rolling. Another factor operating in the same direction is the popularity of the continuous casting process in the United States. Much of the paper is devoted to a description of modern rolling mills and their auxiliary equipment, and the author expresses the view that no very practical equipment for automatic gauge control has yet been evolved. In this connection it may be mentioned that investigations by the British Iron and Steel Research Association[57] have led to the development of two new methods of automatically maintaining constancy of gauge.

The well-known technique whereby metal strip is formed into useful and decorative shapes by cold rolling is rarely described in the literature. A paper covering the practical aspects of the subject, including the machines and tools required, is therefore welcome.[58]

Th Production of Wire. A contribution by Cleaver and Miller[59] to the Institute of Metals Symposium describes present-day wire drawing technique and equipment, special attention being paid to the production of copper and copper alloy wire. Probably the dominating factor in promoting the efficiency which is now achieved has been the introduction of tungsten carbide dies, which permit the use of drawing speeds up to 8,000 ft./min. for medium-fine gauges. It is stated that speeds up to 12,000 ft./min. were achieved in the early 'thirties, but that the tendency subsequently has been to reduce speeds. The drawing speeds of from 3,500–5,000 ft./min., given by Cleaver and Miller as typical of American practice are somewhat at variance with figures mentioned by Kirby[60] at a symposium on Rod and Wire Production Practice held by the American Institute of Mining and Metallurgical Engineers as recently as 1949. He states that wire-drawing speeds in present use at the American Brass Company are about 1,000 ft./min.

Two papers dealing with the more fundamental aspects of wire drawing have also appeared. One of these, by Navarro[61], discusses the influence of the shape of the die on the mechanical properties of the product, copper being the material used for the experiments. The other paper, by Baron and Thompson,[62] embodies an approach to the determination of friction in wire drawing, which is in some degree at least inconsistent with the previous work of Thompson and his former collaborators. While earlier work indicated a linear relationship between the drawing stress and the reduction of area effected by the die, the present paper appears to be based on the assumption that the relationship between these two quantities takes the form of a pronounced curve derived from the true stress-strain curve of the material being drawn. Brass was used for the experiments.

Deep Drawing and Pressing. One of the most informative of the papers in the Institute of Metals Symposium on Cold Working is that by Jevons[63] dealing with the deep drawing and pressing of non-ferrous metals and alloys. After comprehensive descriptions of the various methods in use for the forming of sheet metals and of the presses and tools with which they are performed, the author refers in considerable detail to such auxiliary processes as inter-stage annealing and pickling. He states that of all the non-ferrous metals and alloys which are deep drawn and pressed, brass is used in the greatest quantity, especially "basis" brass containing 62–63% copper and "cartridge" brass with 70% copper. Brasses containing 65% and gilding metals carrying from 80–90% of copper are employed to a lesser extent. The correct selection of grain size in relation to the object being drawn is of considerable importance in the case of brass; the grain size of thin sheet for shallow draws is generally about 0·02–0·03 mm. and for deep draws 0·035–0·045 mm., while heavy sheet for large articles may require a grain size of between 0·045 and 0·065 mm. Beryllium copper, in the solution heat-treated condition, can be handled with substantially the same press tools as those for brass, but as it has a higher rate of work hardening, less severe draws are possible between inter-stage anneals. The behaviour of cupro-nickel (usually the 80 : 20 quality) can be likened to 70 : 30 or even 80 : 20 brass, while the nickel silvers behave in presswork operations in a manner comparable with 63 : 37 brass. Desirable precautions in the deep drawing of copper itself include the selection of a small grain size, usually between 0·01 and 0·02 mm., and the use of quarter-hard instead of fully annealed sheet.

Two experimental investigations concerned with presswork operations are worthy of mention. In the first of these, Chang[64] describes a careful series of tests on the shearing of metal blanks, and reaches the important practical conclusions that for copper and brass the optimum clearance between the punch and die should be between 0 and 10% of the metal thickness, and that the use of "shear" on the punch, so that it cuts progressively around the blank, may reduce the load required by as much as 75%. In various experiments, the shearing stress for soft copper lay between 10·5 and 11·6 tons/sq. in. and for 70 : 30 brass between 15·8 and 18·9 tons/sq. in.

The second of the two experimental investigations mentioned above embodies an attempt by Arbel[65] to correlate the reduction in diameter which can be effected by deep drawing with the true stress-strain properties of the material. It is perhaps unfortunate that this work was carried out in America, where the parabolic relationship between true stress and logarithmic strain is so widely accepted that experimenters tend to adopt it even when it is manifestly at variance with their own data. Thus Arbel refers the deep-drawing properties of annealed 65 : 35 brass to the index of the parabolic stress-strain equation, even though this equation is clearly inconsistent with his experimental curve.

On the more practical side Kirchner and Bevan[66] have assessed the advantages and disadvantages of progressive tools for presswork operations, while the use of polyvinyl chloride plastic punches has been reported from Continental sources.[67] It is claimed that the life of such punches is quite good, up to 15,000 articles being produced in 70 : 30 brass before replacement becomes necessary.

The possibilities and limitations of the American "Marform" deep-drawing process, which is essentially a refinement of the Guerin rubber pad technique, have recently been outlined[68] and it appears that another very similar process, known as the "Hidrew," has been developed.[69]

Spinning and Coining. As it is difficult to find in the technical literature authentic descriptions of these two well established processes, recent papers dealing with both are welcome. Lengbridge[70] assesses metal spinning from the point of view of costs and design. He also describes in considerable detail the technique of the craft and the tools and equipment required. The article by Huguenin[71] on coining is of much the same scope.

The Machining of Metals. Experiments on the machining of copper and mild steel have led Chao and Bisacre[72] to conclude that the most striking effect of increase of cutting speed and feed is on the rate of tool wear, though changes in the geometry of the chip, tool forces, surface finish and build-up on the tool also occur. In many directions, such for instance as the extent of build-up on the tool, the effects due to speed and feed can be treated as functions of the product of speed and feed.

Lubrication. In all the cold-working processes which have been covered by the foregoing notes, lubrication plays an exceedingly important part, and it is therefore a valuable and interesting innovation that a metallurgical body like the Institute of Metals should accept a paper dealing specifically with lubrication. In his contribution to the Symposium on Cold Working, Chisholm[73] gives

recommendations in respect of copper and brass. He mentions additions of metal soaps to improve the film strength of lubricants for the drawing of brass and copper tube, and this is paralleled by a recent patent [74] for the use of chromium oleate in connection with deep drawing, wire drawing and tube drawing operations. For the lubrication of forging dies, Lang[75] recommends a concentrated dispersion of colloidal graphite in water. This is also serviceable as a mould coating when die-casting the copper-base alloys.

Hot Working. Though no important developments appear to have taken place recently in the hot working of copper and its alloys, several reviews of current practice should be brought to the notice of readers. An authoritative description of the hot pressing of brasses is so long overdue that a paper by Danielson[76] covering this subject in considerable detail is exceptionally welcome. Drop forging is dealt with by Hayes,[77] hot rolling by Malborn,[78] and extrusion by France and Thelin,[79] while modified processes for the extrusion[80, 81] and hot piercing[82] of metal billets have been patented. *Protective Atmospheres.* Hotchkiss and Webber[83] have presented a comprehensive review of the various protective and " bright annealing " atmospheres now available to industry. They assess the applicability of each to a number of metals and alloys, including those of copper, and emphasise the well-known precautions against the use of reducing atmospheres when annealing tough pitch copper. Bradley and Hammond[84] describe a newly installed bright annealing furnace for the continuous treatment of copper alloy wire, strip and bar, while an improved continuous bright annealing furnacefor strip, which passes through the heating zoneon edge,has been patented.[85]

To be continued.

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Plating Aluminium with Nickel

By A. G. Thomson

An account is presented of a process for the direct plating of aluminium with nickel which has been developed on a laboratory scale at the Low Temperature Station for Research in Biochemistry and Biophysics. It may be considered that the results are sufficiently encouraging to warrant pilot plant tests.

FOR a number of years the corrosion of aluminium has been studied at the Low Temperature Station for Research in Biochemistry and Biophysics, University of Cambridge and D.S.I.R. The ultimate objective of the investigators is to devise methods of protecting aluminium from corrosion by foods and beverages. This research has led to the development of a direct plating process, which has not yet been tested on a commercial scale but may prove to be of great potential value to various industries in which aluminium or its alloys are extensively employed.

It is well known that aluminium's resistance to corrosion is due to a surface film of oxide or hydroxide. The metal is very low in the electrochemical series and becomes highly reactive when the surface film is removed. As part of the research programme, the investigators desired to dissolve the natural surface film of oxide and replace it with artificially produced films possessing greater resistance to corrosion. This led to a study of etching solutions, which in turn necessitated some means of determining the relative efficiencies of the various etchants used.

Evaluation of Etchants

An iodine test was, therefore, developed in which an aqueous solution of iodine and potassium iodide is used. This solution readily attacks exposed metallic aluminium, but has much less action on the oxide and on parts covered by oxide. Etched and washed specimens are immersed in the solution, the amount of iodine reduced in a given time being a measure of the efficiency of the etchant. Not only are comparative measurements thus obtained, but the test has the further advantage of indicating the time required for satisfactory etching with any given etchant.

The factors which must be considered in the commercial application of etchants are: (1) cost of the chemicals; (2) danger to the operator; (3) life of the solution (which must be reasonably long); (4) time of etching (which must be reasonably short); (5) uniformity of etching; (6) attack on the underlying metal (which must be as low as possible compatible with efficient removal of the film).

Of the numerous etchants tested, trichloroacetic acid was selected as fulfilling these requirements best. Etching with this acid is rapid and leaves a very smooth surface free from pits. One of the difficulties encountered was the precipitation of aluminium hydroxide or basic salts of aluminium, but it was found that this could be much delayed by adding a hydroxy-organic substance which would combine to form a soluble salt. Dextrin was selected as the most suitable substance for this purpose. The best etching solution found at this stage of the investigation consisted of: Dextrin 50 g., water 150 ml., and trichloroacetic acid (melted crystals) to 1 litre. The solution was used at 95° C. and the time of etch was from 1 to 2 minutes, the activity of the solution tending to increase with use.

Direct Plating

There is little doubt that the failure of the usual mineral acid and alkaline etchants to bring the surface of the aluminium into the right condition for direct plating is due to incomplete removal of the surface film, and possibly to the formation of secondary films of the wrong type. Having found an efficient etchant, the investigators naturally became interested in the possibility of direct plating. They therefore experimented with copper sulphate solution and found that copper came down almost instantaneously, forming a thick, loose deposit over the entire surface of the specimen. Subsequent experiments showed that it was possible to control the rate of deposition and obtain adhesive layers of copper or nickel.

Adherent coatings of copper and nickel have been deposited on aluminium both by chemical displacement and by electrodeposition. Coatings deposited by chemical displacement have quite a good appearance after polishing and permit a fairly strong soldered joint. Because of their inevitable porosity and thinness, however, they would probably be unsatisfactory for most commercial purposes, though they might, perhaps, serve as undercoatings for plating further metals. So far as can be seen under a binocular microscope, electro deposition can producecoatings of copper and nickel, free from pores, which arealso thicker and, therefore, preferable for industrial use.

Between etching and plating the metal requires to be washed, but washing in water produces a new water-formed film which, if allowed to become too thick, is very detrimental to the adhesion of deposits. Tests showed that the longer the etched metal is allowed to remain in contact with water, the more unsatisfactory will be the adhesion of an electrodeposit, and that washing in hot water is more detrimental than a cold water wash. When plating by chemical displacement with nickel, which is fairly close to aluminium in the electrochemical table, the surface of the aluminium must be in a more active condition than is required for the plating of copper, which lies higher in the table. The activity of the aluminium can be maintained by washing in acetone, or it can be reduced by means of a controlled wash in water or a mixture of acetone and water.

For plating by electrodeposition a thin water-formed film does not prevent the cathodic discharge of electrons and deposition of another metal under the influence of an applied external current, and is advantageous in one respect, in that it tends to suppress undesirable local cell action. The washing process should be conducted with minimum exposure of the etched surface to air and the metal should not be allowed to dry before immersion in the plating solution. The procedure originally developed for plating with nickel by electrodeposition was to wash the etched metal in acetone, followed by two separate lots of cold water. Plating was then carried out in an agitated solution for 30 minutes at a current density of 50 amp./sq.ft. at room temperature, using a nickel anode. The composition of the solution was as follows :

Nickel sulphate	100 g.	Boric acid	2 g.
Ammonium sulphate	50 g.	Water to 1 litre.	

Difficulties in Way of Commercial Application

At this stage of the investigation a paper entitled " The Etching of Aluminium for Direct Plating with Other Metals "* was presented by J. M. Bryan, B.Sc., Ph.D., at the Silver Jubilee Conference of the Electrodepositors' Technical Society. Considerable interest was manifested, but during the discussion emphasis was placed on two major difficulties which required to be overcome before the method could be commercially applied. The first objection was the high cost of trichloroacetic acid, which was regarded as prohibitive, the other criticism being that the acetone used for washing the etchant would be too dangerous for use on a works scale on account of the fire hazard involved.

During the past 18 months the investigators have been endeavouring to overcome these difficulties and considerable progress has been achieved. The process has been cheapened by bringing down the proportion of trichloroacetic acid in the etchant from 88% to 5%, using as an addition 25% of phosphoric acid, which is relatively inexpensive. Indications are that commercial application is now within sight.

In order to avoid the use of acetone, a number of cheap organic liquids were tried, none of which proved entirely satisfactory. The objection to the use of water for washing is its rapid reaction with etched aluminium, as already mentioned, to reform a surface film with the generation of hydrogen. Eventually it was found that this problem could largely be solved by dipping in nitric acid as an oxidising agent before washing with water, thus preventing the evolution of hydrogen which would otherwise occur, even in the plating solution. By this means improved adhesion of the nickel coating is achieved without the necessity for using acetone. Presumably an oxide or basic nitrate film is formed, but this is not sufficiently thick to inhibit plating, and better adhesion is obtained than after washing in plain water.

* *J. Electrodepositors' Tech. Soc.*, 1950, **26**.

Modified Process

As now modified, the process is as follows :

Standard Alkali Treatment

Na OH	50 g.
Lissapol N	3 g.
Water	to 1 litre

Standard Acid Etch

H Cl (conc.)	2·5 ml.
Trichloroacetic Acid (melted crystals)	50 ml.
H_3PO_4 (S.G. 1·75)	250 ml.
Lactic Acid (S.G. 1·21)	200 ml.
Lissapol N	3 g.
Water	to 1 litre

Etch for 1 minute at 75° C., increasing the time of etching when this becomes necessary.

Nitric Acid Dip

Immerse for 15 seconds in nitric acid (1 : 4), then rinse in water.

Nickel Plating†

$NiSO_4\ 7H_2O$	120 g.
$(NH_4)_2SO_4$	21 g.
K Cl	8 g.
Water	to 1 litre
*p*H	5·6–5·8
Temperature	35° C.

Plate for 1 hour 20 minutes at a current density of 15 amp./sq.ft.

Potential Applications

Various methods for plating aluminium have been developed, such as Travers' process for anodising and then modifying the surface film. Probably the best existing method is the sodium zincate process, originally developed by the American Aluminium Co., which has the great advantage of being cheap. It gives smooth deposits of very good appearance and excellent adhesion. The aluminium is first given an alkali etch followed by a dip in a strong nitric acid. Zinc is then deposited by chemical displacement from alkaline solution, and this is followed by a thin electrodeposit of copper, a coating of nickel, and finally a flash chromium treatment to give brilliance. Despite its apparent complexity the process presents little difficulty once the line of tanks has been set up, some of the dips being quite brief.

The direct plating process developed at the Low Temperature Research Station opens up a new approach, the implications of which cannot as yet be fully assessed. Specimens plated directly with nickel and given a chrome finish have adequate adhesion and, although slightly rougher than samples obtained by the sodium zincate process, are likely to be more resistant to corrosion. A disadvantage of the zincate process is that the deposit is based on a copper undercoating, although attempts are being made to dispense with this for some purposes. The presence of copper is, of course, highly undesirable for many applications associated with food production. A trace of this metal produces rancidity of fats and oils and is very liable to cause corrosion troubles, since copper and nickel, being widely separated in the electrochemical series, set up strong galvanic action when in contact.

† Bath devised by Bullough and Gardam (*J. Electrodepositors' Tech. Soc.* 1947, **22**, 169) for a modified sodium zincate process for plating aluminium with nickel.

From the food manufacturer's standpoint aluminium is much cheaper and more readily fabricated than stainless steel, so that plated aluminium has attractive possibilities. Nickel-plated aluminium equipment might be useful in the dairy industry, where both nickel and aluminium are employed. Difficulties are experienced in cleaning and sterilising aluminium surfaces, which tend to roughen with use and become dull. Nickel-plated aluminium would be more easily cleaned and, if chromium plated, would have a very pleasing appearance. It might also be used with advantage for brewery equipment.

Now that aluminium has become a relatively cheap material, it is being extensively employed in many industries, frequently in alloyed form to give it greater strength. The process has also been applied successfully to the plating of aluminium alloys containing small percentages of magnesium and manganese, such as are used for domestic utensils. There should be important fields for plated aluminium alloys in aircraft, bicycles, and other fields where lightness is important.

A series of tests has been planned to compare the corrosion resistance of directly plated specimens with specimens plated by the sodium zincate process with and without a copper undercoating. This comparison will take some time, but it is obviously necessary before the potentialities of the new process can be evaluated. It is also possible that the new process may provide an approach to the study of the mechanism of adhesion. It has been suggested that the best adhesion is obtained when the lattice structure of the coating metal corresponds with that of aluminium, but at present this theory needs confirmation.

It remains to be seen, therefore, whether the process is a practical proposition, and this, of course, can only be finally decided by trials on a commercial scale. The Low Temperature Research Station has neither the facilities nor the equipment for plating beyond laboratory scale, nor would such a developmental undertaking come normally within the province of the Food Investigation Organisation of the D.S.I.R. Industrialists may consider, however, that the results of the laboratory work are sufficiently encouraging to warrant pilot-scale tests by manufacturers or trade organisations interested in exploring the practical implications of this new process.

Notes on the Metallising of Plastics

Continued from page 290

metallising processes. Like the " shiny spray " methods, they employ Liebig's discovery that oxides and salts can be chemically reduced to provide coatings of metallic silver. The main object of the silvering with this type of process is not brightness of film but electrical conductivity. Obviously, to electroplate a non-metallic material it has to be made conductive, and wet silvering by immersion happens to be a convenient device to provide this initial requirement. Once the conducting film is developed, electroplating can proceed on orthodox lines *via* the acid copper bath. The acid copper bath is preferred because of the rapid chemical attack on microdeposits of silver by cyanide and Rochelle copper solutions. The equipment involved is fairly simple and, were it not for the fundamental problem of adhesion between the silver coat and the plastic, there is little doubt that widespread commercial application would have been possible. Unfortunately, the nature of the silver deposit has been a major obstacle.

For a permanent deposit of electroplate, it is obvious that the silver micro-film which is acting as the connecting link, must be firmly attached to the plastic. As currently operated, the wet silvering processes can only provide a loose attachment, in the absence of either absorption or chemical union of the deposited film with the plastic. This inadequate adhesion of the primary conducting coat is the basic weakness of the technique. It is a condition which is further complicated by the inherent stress in electroplated coatings, the movement of synthetic materials in response to variations of temperature and humidity, and, in some cases, the chemical interference through plasticiser and solvent migration.

On small objects these frustrating influences are less marked. It is possible for the overall grip of a substantial electrodeposit of copper to cancel out the disruptive effect of the contained stress in the plate. Further assistance is frequently provided by undercuts or intricacies of design, but on large pieces, or plain surfaces, the pull of the electro plate virtually guarantees that the deposit will lift away from its plastic base.

Heavy and, therefore, uneconomic undercoats of copper have been suggested, in the vain hope that sheer weight of copper would keep the deposit intact but, unfortunately, the heavier the deposit the greater the stress, and copper plate of the order of 0·003/0·005 in. can only indirectly ensure that what little adhesion did exist between the silver and the plastic is effectively counteracted.

Recent Developments

The limitations of existing processes, and the widespread interest in the commercial possibilities of metalloplastics, has encouraged further research. Few details of current investigations are available but the general trend of them can be noted. In America, some attention has been devoted to conducting plastics which it is hoped will obviate the necessity to build up a primary conducting coat. Early experiments along these lines endeavoured to adulterate the mouldings with conducting metal particles. In order, however, to introduce a sufficiency of the adulterant to ensure overall conductivity, the effect of the impurity is to interfere seriously with the physical properties of the moulding. Low temperature diffusion processes with vapour and solid compounds have proved disappointing, and although a system has been worked out, it is neither as economical nor as efficient as available methods. Metal spraying on thermoplastics is being carefully investigated with the object of obtaining complete or partial fusion between the warm metal particle and the plastic surface.

The most interesting of the British processes appears to be a modified chemical device which effects a controlled reduction of silver on plastic, so that the metal deposit is " alloyed " with the plastic, giving a strength of attachment which is many times stronger than any stress lift that may be provided by even heavy coats of electroplated metal. For two unlike materials such as plastic and metal to be " alloyed " without noticeable interfacial weakness is something of a technical novelty and an important step forward in the search for a true composite structure.

Emergency Specifications For Case-Hardening Steels

NICKEL is one of the important alloying elements used in steelmaking to-day, and shortage of nickel supplies is, therefore, a matter of the utmost concern. A major step towards balancing supply and demand of this element has recently been taken, in the publication by the British Standards Institution of a series of eight new emergency specifications for case-hardening steels (B.S. 970, En. 351 to En. 355 inclusive and En. 361 to En. 363 inclusive, Addendum No. 1 : November, 1951). The magnitude of the saving in nickel effected by these new specifications, details of which are summarised in Table I, is very gratifying. The original series, En. 33 to 39, contained from 2% to 5% Ni. Against this, 2% Ni is the maximum in the new specifications and this amount is called for only in the 85-ton steel ; the lower strengths steels contain from $\frac{1}{2}$% to $1\frac{3}{4}$% Ni.

TABLE I.—SUMMARY OF COMPOSITIONS AND PROPERTIES.

	En. 351	En. 352	En. 353	En. 354	En. 355	En. 361	En. 362	En. 363
Tensile Range—tons/sq. in.	45/55	55/65	65/75	75/85	85 min.	45/55	55/65	65/75
En Steels Replaced	33, 34, 37	35, 36, 325	36, 38	New Range	39	33, 34, 37	35, 36, 325	36, 38
Composition—%								
Carbon	0·20 max.	0·22 max.	0·22 max.	0·22 max.	0·20 max.	0·13/0·17	0·18/0·23	0·22/0·26
Silicon	0·35 max.	0·35 max.	0·35 max.	0·35 max.	0·35 max.	0·35 max.	0·35 max.	0·35 max.
Manganese	0·60/1·00	0·50/1·00	0·50/1·00	0·50/1·00	0·40/0·70	0·70/1·00	0·70/1·00	0·70/1·00
Nickel	0·60/1·00	0·85/1·25	1·00/1·50	1·50/2·00	1·80/2·20	0·40/0·70	0·40/0·70	0·40/0·70
Chromium	0·40/0·80	0·60/1·00	0·75/1·25	0·75/1·25	1·40/1·80	0·55/0·80	0·55/0·80	0·55/0·80
Molybdenum	0·10 max.	0·10 max.	0·08/0·15	0·10/0·20	0·15/0·25	0·08/0·15	0·08/0·15	0·08/0·15
Sulphur	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.
Phosphorous	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.	0·05 max.
Ultimate Tensile Strength tons/sq. in.	45 min.	55 min.	65 min.	75 min.	85 min.	45 min.	55 min.	65 min.
Elongation—%	18 min.	15 min.	12 min.	12 min.	12 min.	18 min.	15 min.	—
Izod Impact—ft. lb.	30 min.	20 min.	20 min.	20 min.	25 min.	25 min.	15 min.	—

The choice of alloying elements in case-hardening steels is limited in at least two directions. First, the effect of alloys on the hardness, wear resistance and microstructure of the high carbon case has to be considered ; secondly, provision must be made for adequate strength, ductility and toughness in the low carbon core. Nickel was early found to have the requisite effects on both these sets of conditions, and case-hardening steel practice has thus been built up around high nickel contents, of the order of 4% to 5%. The En. 33 to 39 series may be looked upon as an early systematisation of existing practice.

The impact of nickel shortages first fell on the blockaded Germans during the 1939–45 war. One of their responses was the development of a 2% Ni-2% Cr-Mo high-duty case-hardening steel, which was later incorporated in B.S. 970, as En. 320. This steel was found to be prone to the formation of carbide net-works in the carburised case, and has never seriously competed with En. 39A and B, the $4\frac{1}{4}$% Ni-Cr and Ni-Cr-Mo steels. En. 325, a 55-ton steel with $1\frac{3}{4}$% Ni-$\frac{1}{2}$% Cr and $\frac{1}{4}$% Mo was another attempt at alloy economy. Comparison with the new 55-ton steels En. 353 and En. 363, will, however, show how much further the process has been carried.

Comparing the old and new series of specifications, it can be estimated that a 50% saving in nickel usage will be effected. Statistics are, unfortunately, not available from which this saving can be translated into the actual tonnage saved. One very important aspect of the new series of steels is that full provision has been made for obtaining the maximum benefit from any alloy scrap steel. The alloy contents permitted are ample to provide the requisite hardenability, and oil quenching is still retained.

Whilst economy in nickel usage is perhaps the most spectacular feature, it is noteworthy that considerable savings of molybdenum, another critical alloying element, have also been achieved. Not all the original alloy case-hardening steels contained molybdenum, but when introduced, 0·25% was the amount usually added. The new steels contain approximately 0·10% Mo, with the exception of the 75- and 85-ton steels, which contain 0·15% and 0·20% Mo respectively. The actual saving of molybdenum cannot be predicted, as it is expected that much of what is used will be obtained from remelted alloy containing scrap, so long as such supplies are available.

The emergency specifications for alloy case-hardening steels are proof of the determined attack which is being made on the problems of alloy economy which are confronting us. The further efforts of the Alloy Steel (Rearmament) Technical Committee will be awaited with eagerness as new milestones on the road to a more settled economical practice.

New Materials Handling Film

ONE of the points which has been repeatedly stressed by Productivity Teams returning from the U.S.A. is the importance which is attached to materials handling in America. The movement and handling of materials increases the cost but adds nothing to the value of the finished product. Even more important is the fact that out-of-date methods of handling absorb an enormous amount of unproductive labour. Perhaps more than any other item of handling equipment, the fork lift truck has been popularly associated with productivity, and an interesting film, " Fork Lightening," dealing with the manufacture and use of this type of truck, was shown recently, in connection with the " National Work Saving Week," by Conveyancer Work Trucks, Ltd., Warrington

NEWS AND ANNOUNCEMENTS

Mond Nickel Fellowship Awards

THE Mond Nickel Fellowships Committee has pleasure in announcing the following awards for 1951 :

MR. J. PRESTON (British Non-Ferrous Metals Research Association) to study, in Great Britain, the United States of America and Canada, specialised methods of production and fabrication of metals with particular reference to powder metallurgical techniques.

MR. P. E. WHITE (Messrs. J. B. & S. Lees, Ltd.) to study the metallurgy and detailed production technique of high-quality strip steels in the United Kingdom, Scandinavia, United States of America and Canada, with particular reference to hardened and tempered steel strip, stainless steels and silicon steels.

MR. P. J. HILL (Public Works Dept. of Western Australia) to study in the United Kingdom the application of research to the development of corrosion resistant metals for use in the mechanical engineering industry

F. B. Richards Equipment Fund

To mark the retirement of Mr. F. B. Richards from the Chairmanship of the Woodall-Duckham Group of Companies after 19 years, service in that capacity, the Directors of the Woodall-Duckham Vertical Retort & Oven Construction Company (1920), Ltd., have arranged to make a grant of £1,000 per annum under a seven year covenant to create an Equipment Fund for the Department of Coal Gas and Fuel Industries with Metallurgy at Leeds University. The capital from the Fund will be utilised by the Livesey Professor at his discretion for the purchase of equipment for his Department. In expressing their deep appreciation of this gift, the University has suggested that the Fund should be known as the " F. B. Richards Equipment Fund." Mr. Richards was for many years a member of the Coal Gas and Fuel Industries (Livesey Professorship) Committee of Leeds University, and took a considerable interest in the success of the Fuel Department.

The British Aluminium Co., Ltd.

THE Head Office address of The British Aluminium Co., Ltd. is now Norfolk House, St. James's Square, London, S.W.1 (Telephone No.: WHItehall 7868). Norfolk House was built in 1938 on the site of the Duke of Norfolk's residence and the Company was to have moved there in September, 1939. At the outbreak of war, however, the building was requisitioned and used later as the Supreme Headquarters of the Allied Expeditionary Force. In the Company's Board Room, General Eisenhower and his Staff planned and launched the Allied operations for the liberation of North Africa and the Continent of Europe.

Students Visits Furnace Works

A PARTY of students from the Borough Polytechnic London, visited the Watford works of Messrs. Wild-Barfield Electric Furnaces, Ltd., to gain a knowledge of the production and the research behind the manufacture of the well-known Wild-Barfield furnaces and high-frequency equipment. All stages of construction were demonstrated including case-making, bricklaying, electrical assembly and testing, and a visit to the research and development laboratories showed the immense amount of behind-the-scenes activity inevitably connected with the solving of customer's metallurgical problems. Special interest was taken in the care and attention given to provide prospective and existing customers with a complete picture of the results to be expected from the heat treatment of their materials in electric furnaces. Messrs. Wild-Barfield Electric Furnaces, Ltd., are prepared to receive requests from suitable schools, societies and associations for the few remaining dates available this winter.

Personal News

MR. JACK GREGORY has been appointed a Director of General Refractories, Ltd., Sheffield. Mr. Gregory, who joined the firm in 1921, was for many years in charge of the company's Worksop Works, and since 1947 has held the position of Works Superintendent in England and Wales.

THE Chief London Representative of Keith Blackman, Ltd., MR. J. C. CAMPBELL, retired last month after 50 years' service. His successor is MR. D. J. AULD.

HEAD, WRIGHTSON & CO., LTD. announce that MR. JOHN LONG, formerly Assistant Manager of their London Office, has been appointed London Manager in succession to MR. SELBY ROBSON. Mr. Robson remains a Director of Head, Wrightson & Co., Ltd. and of Head Wrightson Processes, Ltd., resident in London.

THE Fulmer Research Institute of Stoke Poges, Bucks., announce that SIR DAVID BRUNT, M.A., Sc.D., F.R.S., has been appointed to the Board of Directors.

MR. C. S. GILL has resigned from the Board of Davy & United Engineering Co., Ltd.

MR. J. C. CARR, C.B.E. has been appointed to the Board of Thomas Summerson & Sons, Ltd., the Railway Siding Engineers, and their subsidiary companies, on his retirement from the post of Principal Assistant Secretary in the Ministry of Supply.

THE RT. HON. THE EARL DE LA WARR has resigned his Directorship of The Brush Electrical Engineering Co., Ltd., on his appointment as Postmaster General. MR. IAN THOMAS MORROW, late of Robson, Morrow & Co., has been appointed to the Board.

Obituary

WE regret to record the death, on November 22nd, of Mr. George Harry Rogers, B.Sc., M.Inst. Met., F.I.M. Mr. Rogers, who was in his 62nd year, spent the whole of his working life in the service of the Metals Division of Imperial Chemical Industries, Ltd. and its predecessors. Joining the King's Norton Metal Co. in 1904, he became Assistant Works Manager nine years later. Another spell at King's Norton—this time as Works Manager—broke twenty-five years' service at the Division's Kynoch Works, where he ultimately became production manager for strip and sheet manufacture. In 1946 he was appointed Regional Production Manager of the Division's three factories in South Wales.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Resistance Thermometer Controller

THE new Sunvic Resistance Thermometer Controller type RT.2, now in production, has been designed to overcome the effect of the present wide variations of mains voltage and frequency on the temperature control of electrically heated furnaces. These variations represent one of the most difficult problems in furnace temperature control. They are overcome by the incorporation of the well-known Sunvic Energy Regulator principle in the output circuit of the controller. This principle compensates for the change of control point which would otherwise accompany change of supply voltage. This feature permits the use of a simple, and hence cheaper, furnace since, one source of control point movement being eliminated, a wider proportional control band may be used, creating conditions in which temperature stability of the furnace is much easier to achieve.

The controller is arranged for mounting in standard P.O. racks, the front plate being half standard rack size. A neon lamp is provided to indicate the action of the load contacts, and a bridge balancing potentiometer and sensitivity control are available on the front of the case. Two industrial type vacuum valves are used and no electrolytic capacitors are employed. The RT.2 Controller is designed to give accurate temperature control of creep test furnaces, muffle furnaces and electric ovens.

Sunvic Controls, Ltd., 10, *Essex Street, London, W.C.2.*

Bronx 60-ton All-Steel Press Brake

AN all-steel welded press brake of capacity 8 ft. 0 in. × $\frac{1}{8}$ in. mild steel and 60-ton normal rated pressure, embodying a number of refinements expressly for the purpose of easing the work of the operator and the tool setter is made by the Bronx Engineering Co. Ltd.

The frame of the machine itself is of massive all-steel welded and annealed construction, and is guaranteed unbreakable. The drive is taken by V-belts from the main 5 h.p. high slip motor to a pulley integral with flywheel clutch and lugged brake, all of which are mounted on the first motion shaft carried in anti-friction bearings. From this first motion shaft the drive is taken with steel helical gears to the second motion shaft, carried on ball and roller bearings. Double reduction pinions at each end of the frame take the final drive to the twin eccentric shafts which are machined from solid steel forgings. These are carried in the steel crown in large diameter phosphor bronze bearings. Cast iron pitmans fitted with large phosphor bronze worm wheels and high tensile spherical ended screws transmit the power to the steel plate top beam.

A ram stop mechanism is fitted, so that, at will, the press will single stroke, coming to rest automatically at top dead centre. By moving a lever the mechanism is disconnected and the machine works as a normal press brake. The operator has perfect control over the top beam and inching can be carried out with the patent multi-plate dry clutch at any position of the cycle. Motorised power adjustment by means of a 2 h.p. motor is provided with safety limit switches for the top beam. This motor for operating the top beam, for die-opening adjustment, is mounted on a bracket integral with the left-hand side connecting rod. The limit switches are incorporated to prevent over-run of the top beam when being moved, either up or down, for tool adjustment.

In the event of a "stick-up," that is, when the top beam tool sticks just below bottom centre due to incorrect tool setting, the beam can be easily released by barring up, provision being made for this by cast-on lugs.

Bottom dead centre is indicated at the front of the machine by a large dial let into the top crown. Counter indicators are provided for each connecting rod to facilitate tool changing. Other particulars of this machine are: distance between columns 8 ft. 4 in.; overall length over beam 10 ft. 0 in.; strokes per minute 30; stroke 3 in.; ram adjustment 6 in.

Bronx Engineering Co. Ltd., Dudley Road, Lye, Stourbridge, Worcs.

A New Nickel Chromium Alloy

IN an effort to produce an alloy with a high resistivity and a low and stable temperature coefficient, British Driver–Harris advise us that they are developing a nickel chromium alloy which will have the following properties:—

Resistivity—800 ohms/circular mil-foot.

Temperature Coefficient— ± ·00002/° C. (20°–100° C.)

Although the manufacturers are not in production, samples are available in sizes down to 0·001 in. As it is believed that an alloy with these properties would fulfil very useful applications, the Company invite all those interested to communicate with their Technical Department.

British Driver–Harris Company Ltd., Manchester.

Staffa Single Spindle Screwing Machine

As an addition to their well-established range of products, Chamberlain Industries Ltd., now introduce their Staffa $\frac{3}{8}$ in. single spindle screwing machine, a compact self-contained and robust machine which is highly serviceable for the accurate high-speed threading of bolts, pipes and small components. The pedestal of the machine is of fabricated construction, comprising two side panels, pressed-out from mild steel sheets, which form an integral part of the cutting lubricant and swarf cradle. At the front and rear, removable panels allow complete access to the interior.

A hollow spindle, upon which a rotating die head can be fitted, runs in self-lubricating Oilite bearings with the thrust washers of the same material. A small gear pump, housed with a reservoir, pumps the cutting lubricant through the hollow spindle to wash the die head clean of swarf, and keep both sides of the chasers clean. To seal any oil leak at the end of the spindle, an oil retainer is incorporated in the rear bearing housing. The spindle and lubricant flow control fittings are totally enclosed at the top of the machine by a removable push-on cover. The die head itself is enclosed by a

hinged cover, ensuring cleanliness and complete protection for the operator against lubricant and revolving components.

The carriage assembly comprises a flat top table, to which any form of self-centring vice or work-holding fixture may be mounted. Two tubular guide bars, running the length of the cradle, provide the slide bed and are extended on either side of the front bearing housing. Oilite bushes provide for the frictional wear caused by the sliding movement of the carriage. The guide bars maintain the correct alignment of the table top horizontally and vertically with the spindle. Coupled to a simple linkage arrangement positioned at the rear end of the cradle, the forward and return movement of the carriage assembly is controlled by the action of a lever swinging through an arc of 90°. No cams, gears, or racks and pinions are used, and the danger of swarf clogging such components is thus eliminated.

To predetermine the length of thread to be cut, two screwed shafts with trip adjusting nuts are housed in the tubular slide bars. The nuts are adjusted inside the slide bars by means of two control knobs at the front end of the cradle. These knobs are graduated with markings to indicate the length of the table movement. The carriage is devoid of springs or return weights to avoid drag on the work, resulting in deformation of the thread. The forward and return action of the carriage registering against the trip nuts ensures automatic opening and closing of the diehead. In case of emergency and as a safety precaution, the operator can open the diehead by pressing the control knob positioned at the front end of the cradle.

The drive of the spindle is through a single V-belt running on an adjustable cone pulley. In the base of the pedestal, and mounted on sloping rails, is a $\frac{3}{4}$ h.p. protected electric motor; adjustment for belt tension is readily made by moving the motor up or down the rails.

The capacity of the machine, varying according to the type of die head used, is $\frac{3}{16}$ in. to $\frac{3}{8}$ in. B.S. Whit., $\frac{3}{16}$ in. to $\frac{1}{4}$ in. B.S. Fine, $\frac{1}{4}$ in. Gas or $\frac{1}{2}$ in. Conduit. The travel of the carriage is 9 in., and the speed range 266 to 1,007 r.p.m. The floor space required is 3 ft. by 1 ft.

*Chamberlain Industries Ltd., Staffa Road, Leyton, London, E.*10.

Electronic Relay

THE high inherent accuracy of the bimetal thermostat, contact thermometer or toluene regulator can be realised only if the contacts associated with them have no snap-action. Any such action, which is designed to prevent arcing at the contacts, represents a temperature differential at the control point, and is hence a source of error. By reducing the current in the contacts to a negligible value the new Sunvic Electronic Relay type ED.2 removes the need for a snap-action and permits their use under optimum conditions. In addition, the incorporation of a thermally operated energy regulator unit in the output of the relay renders it responsive to the mean current passing through the contacts. If, therefore, vibration is introduced at the thermostat contacts, there is a small range of temperature over which proportional control is provided, greatly improving the accuracy with which the controlled temperature is maintained. This facility is the subject of a provisional patent.

The relay is designed to operate from 100 to 250 volt A.C. and has a consumption of approximately 15 watt The current in the sensitive contacts is of the order o ten microamps while the output contacts are rated a 9 amps 250 volts A.C. The relay may be vertically o horizontally mounted, and the case is finished i Hammer Grey cellulose enamel.

Sunvic Controls, Ltd., 10, *Essex Street, London, W.C.*2

R. & R. Precision Lap-Grinding Machin

RUDKIN & RILEY LTD. have added a precision lap-grinding machine to their range of die-shop equipment. This R. & R. type 2020/1 grinder, will accommodate round and shaped laps up to $\frac{3}{4}$ in. dia., and of unlimited length. The machine occupies bench space of 42 in. × 23 in.

The 6 in. dia. grinding wheels are on the spindle extensions of a totally-enclosed $\frac{1}{4}$ h.p. motor mounted on a saddle which has transverse adjustment on the bed by means of a handwheel and screw. The two grinding wheels are totally enclosed apart from small apertures in the guards to allow entry of the work-piece.

The three-jaw chuck on the workhead is carried on a hollow spindle which permits handling of the long laps, and is driven by a $\frac{1}{8}$ h.p. motor. The workhead is mounted on a graduated swivelling base which enables tapered laps up to 50° included angle to be ground. An indexing device is also fitted to the workhead spindle to enable shaped laps, such as squares, hexagons, etc., to be ground.

The whole lap carriage is adjustable longitudinally by means of the handwheel-operated chain and sprocket motion; this enables a smooth reciprocating action to be imparted to the lap during grinding. For grinding shaped laps, the grinding turn-table swivels through 90° to present the face of the wheel to the work, and by this means the lines set up on the lap in the grinding process are at right angles to the lap direction—this renders the compound more effective when lapping dies. Sharp corners on shaped laps are quickly maintained by a slight skim with the grinding wheel when the lap shows signs of wear.

Rudkin & Riley Ltd., Cyprus Road, Aylestone Leicester.

Warren Morrison Valve

A VALVE designed to handle chemicals, corrosives, slurries, etc., is made by Warren, Morrison Ltd. It is a development of the well-known laboratory clip principle. The renewable, flexible inner tube may be one of several grades of rubber. It gives no resistance to flow, having a "straight-through" bore. It has end flanges which form the pipe joints and ensure the isolation of fluid from the metal body.

The body may be of aluminium, iron, bronze, etc., and with the closing mechanism is designed to support the tube which, therefore, becomes a flexible lining and does not have to withstand the fluid pressure over more than very small areas. On $1\frac{1}{2}$ in. and smaller valves, closing is from the top only, and on 2 in. and larger valves closing is from top and bottom of the tube.

Warren, Morrison Ltd., 29, *Bury Street, St. James's, London, S.W.*1.

CURRENT LITERATURE

Book Reviews

METALLURGICAL THERMOCHEMISTRY

By O. Kubaschewski and E. Ll. Evans. pp. xi + 368, 103 illustrations. Butterworth-Springer, Ltd., London, 1951. 35s. (by post, 36s.).

In recent years it has been increasingly evident that developments in the production of alloys and metals follow from fundamental knowledge of the reactions involved. One very powerful method of obtaining such information is by use of the principles of chemical thermodynamics. This approach is followed by the authors who use the term "Thermochemistry" in its widest sense to include not only the consideration of heats of formation and reaction but also free energy and entropy changes. While it is assumed that the user of the book will have at least an acquaintance with chemical thermodynamics, the basic principles are clearly outlined in the first part of the book and the argument is logical and self-contained. Signs and symbols are given at the outset together with the necessary physical constants and their accepted values

A section on calorimetry includes a detailed survey of calorimetric methods in which principles are emphasised and details of special techniques given. Here some mention might, perhaps, have been made of the use of thermistors in the determination of small temperature or heat changes. In addition to the determination of heats of reaction, methods of determining heat of solution, fusion and transformation are included together with methods of determining heat capacities. This section of the book also includes the use of E.M.F. methods to derive thermochemical data and the experimental methods used to obtain such data for heterogeneous equilibria involving gases. The advantages and disadvantages of different methods are discussed and assessments made of the magnitude of possible errors.

A section on the estimation of thermochemical data includes methods of calculation and estimation of the heat capacities of gases, liquids and solids and of heat and entropy changes in phase transformation and compound formation. Such methods are of considerable value in checking experimental data and for estimating values not capable of experimental determination. Experience is necessary to enable the best choice of method to be made and the authors' estimates of the accuracy of the methods should be of help in this connection.

The latter part of the book is mainly devoted to a collection of thermochemical data. A hundred pages of tables include heats of formation, fusion, evaporation and transformation, entropies and vapour pressures, for compounds and elements of interest to the metallurgist, including intermetallic compounds. Free energies of reactions are also tabulated and thermochemical data for some binary metal systems with continuous and limited solution. The authors have made a selection of the most accurate values and in some cases have recalculated values in the light of recent information. Full references to these tables are given and the authors are to be congratulated on their compilation of data likely to be of great value to both the research and process metallurgist.

The book includes a section of examples of the thermochemical treatment of metallurgical problems. Though the number of examples is small they appear to have been chosen with care and should be of value in the indication of possible methods of approach to a problem.

The list of references given is large and includes a list of relevant papers published while the book was in print. While, as the authors state, the book is designed for the research and process metallurgist, parts will also be of value to the advanced student reading for an honours degree in metallurgy. Much will also be of interest to physical chemists in indicating the extension of chemical thermodynamics to metallurgy. The book is well produced and the price is not excessive.

W. R. Moore.

TYPICAL MICROSTRUCTURES OF CAST IRON

Published by the British Cast Iron Research Association, Alvechurch, Birmingham, 1951. 54 pp., 41 illustrations. 21s. net (15s. to Members of the Association).

Attempts have been made in the past to illustrate the structures of metals and alloys as viewed under the microscope, notably the comprehensive collection known as the "Atlas Metallographicus," by Hanemann and Schrader, based on work carried out at the Technische Hochschule, Berlin, before the last war. Less comprehensive but more specialised collections have been attempted with varying degrees of success and quality of reproduction both in America and this country. In 1933 and 1936, the British Cast Iron Research Association collaborated with the Institute of British Foundrymen in preparing two small booklets, illustrating some microstructures of cast iron, for the use of foundrymen.

The present work is intended to provide, for reference and comparison, a series of photomicrographs of the principal types of cast iron normally encountered by the metallurgist and engineer in their daily work, at magnifications to which they are accustomed. The book is provided with a technical introduction, which indicates the dependence of the structure and properties of cast iron on cooling rate and composition, and considers separately the mode of occurrence and appearance of the constituents of cast iron.

The Association has previously issued methods of preparing specimens for microscopic examination; the last, by H. Morrogh, in 1938, formed the basis of a comprehensive and beautifully illustrated account of the preparation of metallographic specimens presented to the Iron and Steel Institute. The text of the present work does not, therefore, include any account of polishing and etching methods, although the etching reagent used is stated beside each photomicrograph. The 41 illustrations are printed on large size paper ($11\frac{1}{4} \times 8\frac{1}{2}$ in.) and are accompanied in each case by an explanatory note on the facing page. They are grouped under constituents of cast iron, unalloyed cast irons, special and alloy cast irons, and malleable cast irons, and the work is concluded with a few photomicrographs illus-

trating some of the commoner defects encountered in iron castings.

The work of Morrogh on the metallography of cast iron has led one to expect a high standard of excellence in photomicrographs emanating from the laboratories of the B.C.I.R.A. and Mr. D. Marles, who undertook the choice of specimens and their preparation and polishing, together with the descriptive text which accompanies them is to be congratulated on maintaining that reputation. Finally, one must join with Dr. J. G. Pearce, the Director of the Association, in the tribute he pays in the Foreword to the blockmakers and printers "for making every effort to ensure as faithful a degree of reproduction as is possible in the half-tone process."

Trade Publications

ALTHOUGH neither silver nor its principal alloys offer the engineer great mechanical strength or hardness, silver has many other interesting properties and some unique advantages. It has the highest electrical and thermal conductivities of all metals, and the highest optical reflectivity. It is not subject to oxidation at normal or elevated temperatures, and is resistant to attack by many industrial chemicals. In addition it is readily cold worked by any of the commercial processes into a very wide range of manufactured forms. These characteristics, singly or in combination, account for the extensive use of silver for specialised products throughout the electrical, chemical and general engineering industries. Johnson, Matthey & Co., Ltd., Hatton Garden, London, E.C.1., have recently issued "Data Sheet 2010," a 16-page booklet entitled "Silver: Engineering Properties and Uses," which is intended to serve as a general guide to the properties, uses and availability of silver and its principal alloys.

APPROACHING the 150th anniversary of its foundation is the firm of George Fischer, Ltd., makers of fittings, wheels, machines, malleable iron, grey iron and steel castings, whose main works are situated at Schaffhausen, Switzerland. The George Fischer Works has recently published an excellent 26-page volume outlining the development of the company. It is understood that this is one of a series of publications to be issued in honour of the occasion. The story of these last 150 years is told in a most interesting manner, and with the aid of the beautiful illustrations, which are worthy of comment in an age when the value of good illustrations is becoming increasingly appreciated, an insight is given into the many-sided activities of the Company, from its up-to-date workshops to the welfare schemes for its employees.

A NEW edition of the British Aluminium Company's publication, "Aluminium in the Chemical and Food Industries," has recently appeared. It has been produced as a successor to an earlier volume of the same name, issued in 1936 under the auspices of the International Aluminium Bureau. The book is divided into three chapters, the first dealing with the general properties of aluminium and its alloys, including such topics as fabrication, jointing, surface treatment, etc. The middle, and largest, chapter gives details of the action of various substances on aluminium and its alloys, whilst methods of testing corrosion resistance are covered in Chapter III. Owing to the limited number copies are only available to firms and reference libraries.

VARIOUS authorities have shown that the theoretical maximum melting efficiency of the cold-blast cupola is between 40% and 50% of the heat available in the coke. Thus if present practice could be improved by 10%, the effective saving in the foundry would be likely to be of the order of 25–30%—an economy well worth achieving. Such economy—including in the term the melting of improved metal with consequent reduction of scrap castings—could be attained in many foundries by intelligent application of instrumentation. The operative word is "intelligent"; the installation of a meter will produce no improvement at all—it must be used intelligently. To this end the Metronic Instrument Co., Ltd., Ettingshall, Wolverhampton, have issued a booklet, "How to Read the Cupola Meter," which shows the information given by this instrument (air flow and pressure) in relation to the operation of the cupola in normal and abnormal circumstances.

UNTIL recently, lead was the only metallic cable sheathing in general use. Present-day economic and technical considerations have, however, shown a need for alternative forms of sheathing and in recent years, many materials, both metallic and non-metallic, have been investigated. Aluminium has obvious advantages. The metal costs less, volume for volume, than lead and its weight is considerably lower; as a cable sheath it is stronger and harder, yet allows adequate flexibility; it also possesses high conductivity and can be effectively protected against corrosion. In "Aluminium-Sheathed Cables," a recent publication by British Insulated Callender's Cables, Ltd., the salient features of such cables are presented. Copies are available on request from the Company at 21, Bloomsbury Street, London, W.C.1., or from Branch Offices.

WE have received from the Instrument Division of Dawe Instruments Ltd., a new six-page illustrated brochure summarising the wide range of electronic measuring equipment manufactured by the Company. This includes resistors, condensers, inductors, A.F. oscillators and D.C. sources, meters and indicators, frequency and waveform instruments, gauges, stroboscopes and speed measuring equipment, vibration and noise meters, moisture determination apparatus, and high speed photographic flash bulbs. Copies may be obtained from Dawe Instruments Ltd., 130, Uxbridge Road, Hanwell, London, W.7.

Books Received

"The Non-Destructive Testing of Metals," by R. F. Hanstock, Ph.D., F.Inst.P. Institute of Metals Monograph and Report Series No. 10. 163 pp. inc. indices. London, 1951. The Institute of Metals. 21s.

"Experimental Spectroscopy," by Ralph A. Sawyer, Ph.D., Sc.D. 2nd Edition. 358 pp. inc. indices. London, 1951. Chapman and Hall Ltd. 30s. net.

"Metallurgische Verarbeitung von Altmetallen und Rückstanden" Band II: "Altrometalle," by Edmund R. Thews. 290 pp. inc. index. München, 1951. Carl Hanser Verlag. 31·50 DM.

"Man and the Chemical Elements," by J. Newton Friend, D.Sc. (Birmingham), Ph.D. (Würz), F.R.I.C. 354 pp. inc. indices. London, 1951. Charles Griffin and Co. Ltd. 27s. 6d. net.

METALLURGICAL DIGEST

Isothermal Transformation of Austenite

CONTINUING an earlier investigation, several ternary alloy steels of low alloy content, and with ordinary amounts of silicon, manganese, phosphorus and sulphur, have been examined. In most of them cementite was the only carbide constituent present.

The isothermal transformation rates and structures obtained were studied and TTT diagrams obtained. The carbide constituent was isolated electrolytically, X-rayed and analysed: (*a*) after tempering of martensite at constant temperature for different periods; (*b*) in pearlite, as freshly formed at the nose temperature of its 99% transformation curve, and after holding for different periods at that temperature; and (*c*) in bainite, as freshly formed near the nose temperature and after subsequent holding at different temperatures. The main results obtained were as follows:—

(1) At equilibrium, so-called ortho-equilibrium, just below A_1, the ratio between the contents of the alloying element in cementite and in ferrite was:

Cu, Al, Si, P	zero
Co	0·23 : 1
Co	about 0·3 : 1
W	about 2 : 1
Mo	about 8 : 1
Mn	11·4 : 1
Cr	28 : 1

The variation of this ratio with temperature was not determined.

In vanadium steels, even of low alloy content, vanadium carbide is present at ortho-equilibrium below A_1. Hence the vanadium content of cementite at equilibrium is very low and has not been determined.

(2) In most cases of just completed bainite transformation, when its carbide was isolated without appreciable decomposition, its alloy content was very near to the so-called para-equilibrium value—i.e., the value obtained when the ratio of the alloying element to iron is the same as in the austenite. Probably, in all low alloy steels, the bainite as just formed consists of para-ferrite and para-cementite.

(3) The cementite formed at an early stage of tempering of martensite has the para-composition or nearly so.

(4) On isothermal annealing of bainite, or tempering of martensite, the composition of the cementite gradually approached ortho-equilibrium, at a rate depending on the alloying element, concentration, transformation temperature (for bainite) and annealing temperature. The rate was rather rapid for silicon and vanadium, moderate for cobalt, nickel and chromium, and slow for manganese. For molybdenum it was rapid at first and slow towards the end.

(5) The mechanism of pearlite transformation was found not to be the same in all steels examined. The results, being too few to admit of generalization, were as follows:

(*a*) Pearlite just formed isothermally at the nose temperature in nickel, cobalt, silicon and manganese steels contained para-cementite, which gradually changed towards ortho-cementite on continued holding at the same temperature.

(*b*) In molybdenum steels, two kinds of pearlite were found: in an upper range a so-called wrinkled pearlite containing so-called wrinkled ferrite, and in a lower one a so-called smooth pearlite. In the wrinkled pearlite, the ferrite constituent is believed to be ortho-ferrite and the cementite, at the earliest stage analysed, had a composition between para- and ortho-equilibrium, changing with time towards the latter. The smooth pearlite is believed to be para-pearlite.

Even at rather low molybdenum contents there appears, at high temperatures of transformation, an additional eutectoid containing ferrite and double carbide $(Fe, Mo)_{23}C_6$.

(*c*) In one chromium steel examined, the cementite of pearlite just formed was intermediate between para- and ortho-composition. In another chromium steel, there was an upper pearlite of approximately ortho-, and a lower pearlite of para-composition, both as just formed.

(6) In the hypo-eutectoid molybdenum steels examined, the pro-eutectoid ferrite, at higher temperatures of formation, had an ordinary appearance, but, from 675° C. downwards, showed a characteristic wrinkled etch relief. It often had a prickly contour towards the austenite. In the same steels, the pro-bainite ferrite formed in the upper bainite region, and to some extent at the same temperature as the wrinkled ferrite, was not wrinkled. The 1% transformation curves of the two ferrites are independent and intersect. Since the pro-bainite ferrite is probably para-ferrite, like the bainite ferrite, the pro-pearlite ferrite is assumed to be ortho-ferrite, with higher molybdenum content than that of the austenite. An explanation of the wrinkled structure has been suggested on the basis of a secondary carbon diffusion in the adjoining austenite during precipitation.

(7) In several hypo-eutectoid molybdenum, chromium and nickel steels, after some ferrite has been isothermally precipitated, holding the specimen at the same temperature or some other temperature below A_1 was found to cause carbide precipitation in the ferrite, in different ways:

(*a*) As isolated particles at favoured sites in the ferrite, such as ferrite and old austenite grain boundaries.

(*b*) Finely dispersed within the ferrite grains.

(*c*) At the phase boundary ferrite-austenite.

Hence, the idea would seem to be verified that the compositions of a precipitated phase, and of the adjoining layer of the mother phase, are governed by the solubility surfaces of the equilibrium diagram as extrapolated to the precipitation temperature. The ferrite being in equilibrium with austenite is supersaturated with respect to carbide below A_1.

(8) A special form of eutectoid, called F eutectoid, has been observed in connection with phase boundary carbide. It is assumed that the latter is nucleated in the ferrite and grows along the interface and into the ferrite and the austenite as well. The carbon enrichment in the adjoining austenite thus being consumed, the ferrite can keep on growing into the austenite, fresh carbide being later nucleated at a new position of the boundary, etc.

(9) Possibly, bainite is an F eutectoid of para-ferrite and para-cementite. Owing to its low temperature of

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formation, the para-ferrite ought to be appreciably supersaturated with respect to para-cementite.

(10) In certain steels—e.g., silicon steels, the bainite structure is exceedingly fine. An attempt has been made to explain this fact as a result of insolubility of silicon in ortho-cementite and rapid ortho-adjustment of the para-cementite.

High Chromium Steels Resist Attack By Liquid Bismuth Alloys

By John L. Everhart and Edgar L. Van Nuis

IN many industrial processes efficiency is increased by elevating temperatures, one of the limiting factors being the manner of heating the reaction vessel or of transferring heat from one piece of equipment to another. Steam is commonly used at relatively low temperatures; at somewhat higher temperatures organic liquids or molten salts are employed; investigations now indicate that metals will be employed to extend the useful range of liquid media to 800°–1,400° F. (425°–760° C.) and possibly to higher temperatures.

Liquid metals have certain definite advantages as heat transfer media; they have high thermal conductivities; their heat transfer coefficients are among the highest known, being many times those of the organic liquids; and they do not decompose, a factor which limits the range of the organic compounds; most of the metals under consideration have high boiling points, thus permitting operation of equipment at relatively low pressures; electromagnetic pumping is possible, simplifying the pumping problem considerably by eliminating moving parts and packing. On the other hand, these liquid metals may be quite corrosive toward constructional materials.

Among the metals considered are mercury, sodium-potassium alloys, and bismuth alloys. Mercury has been used in boilers since 1922, and several new installations have been made recently. The maximum economical service temperature is limited to about 1,000° F. (540° C.) because of its low boiling point. The sodium-potassium alloys have low densities and their heat transfer properties are attractive. They have been used successfully in a number of heat transfer applications. These alloys, however, are hazardous in the event of equipment failure.

The properties of several of the bismuth alloys are given in the accompanying table. Among the desirable features are the low melting points, particularly of the bismuth-lead-tin and bismuth-indium-lead alloys. These alloys are potentially useful over a wide range of temperature. In comparison with the sodium-potassium alloys they have the disadvantage of much higher density.

The use of bismuth alloys in heat transfer applications depends on the selection of suitable constructional materials to contain them. Investigations of the rate of attack of bismuth lead alloys on such materials indicates that nickel and high nickel alloys, copper and copper alloys, and cobalt alloys are unsuitable as containers. Among the steels, the high chromium alloys are, in general, more resistant than the nickel-chromium materials.

Bismuth alloys, because of their low melting and high boiling points, can be used over a wide temperature range; they have been substituted for oil in heat exchangers because of their superior conductivity and heat transfer properties. Since it is possible to achieve extremely close temperature control by using a circulating liquid metal as the heating medium, these alloys will probably see service in the heating of high temperature reaction vessels, either to add or remove heat, depending upon the requirements of the process. Other uses which have been suggested are the control of die temperatures in die-casting machines; control of mould temperatures in glass making; fractional distillation of metallic vapours; and the reheating of steam.

From *Materials and Methods*, **34**, 112–114

PROPERTIES OF THE LOW MELTING ALLOYS

	Bismuth-Lead	Bismuth-Lead-Tin	Bismuth-Indium-Lead
Composition, % Bismuth	55·5	52	52·3
Lead	44·5	32	25·8
Tin	—	16	—
Indium	—	—	21·9
Density, lb./cu. in.			
Solid 68 F.	0·379	0·352	0·344
Liquid	0·371 (200 F.)	0·346 (212 F.)	0·337 (160 F.)
Melting Point, F.	255	203	158
Boiling Point, F.	3038	—	—
Thermal Cond., Btu./hr./sq. ft./ft./°F.			
Solid	5·8 (100 F.)	—	—
Liquid	6·3 (570 F.)	—	—
Specific Ht., Btu./lb./°F.	0·035 (290–680 F.)	—	—
Viscosity-Centipoises Liquid	2·28 (275 F.)	2·58 (212 F.)	2·29 (212 F.)
Heat Transfer Coef., Btu./hr./°F./sq. ft.	3700 (10 ft./sec.)	—	—
Expansion, % on Freezing (Contraction)	−1·43	−1·53	−1·72
In Solid State	0·77	1·54	0

Micrographic Etchant

By J. Herenguel and M. Scheidecker

ACCORDING to Lacombe, precipitation in aluminium-magnesium alloys can be detected by a combination of mechanical (if necessary) and electrolytic polishing followed by anodic oxidation, the latter resulting in preferential attack on the grain boundaries if the precipitated phase is present in them. This attack is reflected in the structure of the oxide film. The surface of the oxide film is examined under very oblique illumination.

The authors have now discovered that grain boundary precipitation in aluminium-magnesium and also in aluminium-copper alloys can be successfully detected if the specimen is subjected to a combined polishing and etching treatment in a bath used for the chemical polishing of aluminium. The bath contains 85–75% phosphoric acid (60° Bé) and 5–10% fuming nitric acid, and it is used, at a temperature of 85°–105° C. the time of treatment being about one minute. After removal from the bath, the specimens are at once thoroughly mixed with water and dried. This method of etching has been found to be very sensitive in revealing the different degrees of grain boundary as well as intergranular precipitation,

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LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS

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The Use of the Spekker Photoelectric Absorptiometer for Precision Measurements

By F. F. Pollak, D.Sc. A.R.I.C., and J. W. Nicholas, M.R.C.S., L.R.C.P.

The Critall Manufacturing Co. Ltd.

An account is given of the H 602 Spekker absorptiometer with mercury vapour lamp. The technique of its use is discussed and refinements are described enabling it to be used for work of greater precision. The use and preparation of monochromatic filters, the recalibration of the drum scale in true density, and the determination of the true optical depth of glass cells are described. A determination of the molecular extinction of potassium chromate, and a method for the simultaneous determination of chromium and manganese in steel are described as examples of the accuracy which can be obtained.

INSTRUMENTAL methods of analysis are being increasingly used in metallurgical work because of their rapidity. At present, their accuracy cannot compare with orthodox techniques, but there is no reason to believe that, in the case of absorptiometry, this limitation is inherent in the method. Payne[1] gives ± 2% as the normal accuracy of metallurgical determinations on the Spekker, and while this might be satisfactory in the case of an impurity present in amounts of much less than 1% it would hardly suffice for a major constituent. It is the purpose of the present communication to show that, with proper care, absorptiometric determinations on the Spekker can be quite as reliable as conventional procedures.

The absorption of monochromatic light by a solution is an intrinsic function of the ions or molecules it contains; it will, therefore, be a measure of the number of these, and will depend on both the optical depth of the solution and on its concentration. Bouguer and, later, Lambert and Beer have investigated the manner in which absorbing solutions behave, and their conclusions are summarised in what is usually known as the Lambert-Beer law, $d = kcl$, where d is the density (logarithm of the reciprocal of transmission), k is a constant typical of the substance under examination, c is the concentration, and l is the optical depth of the solution.

If c is expressed in mol/litre, and l in cm., the constant will be the molecular extinction coefficient ϵ; if c is in % w/v, and l in cm. the constant is the specific extinction coefficient $E^{1\%}_{1cm}$. Both ϵ and $E^{1\%}_{1cm}$ are characteristic of a given substance at a given wavelength. Density in the above formulae is, of course, relative to the solvent or other blank. There is a certain amount of confusion about nomenclature in the literature, but the above is the usage recommended by the Society of Public Analysts and other Analytical Chemists[2] which has now become general in England. It should be noted that United States terminology has recently diverged sharply from European. The new American terms and symbols are explained by Mellon[3] and Gibson.[4]

It is frequently found that absorptiometric calibration graphs are curved, and this is referred to as deviation from Beer's law. In fact this is usually not so, but is a mixed light error. States and Anderson[5] have shown that, with a substance obeying Beer's law, a curved calibration graph is to be expected if any light is present other than the desired wavelength, with the sole exception of the special case where the absorption of the stray light is equal to that at the desired wavelength. Less frequently, true deviations from Beer's law occur due to some chemical change (e.g., dissociation or complex ion formation) with different extinction coefficients at different concentrations; here again the graph will be curved. A linear relationship is obviously desirable for convenience in calculation, as it is then possible to dispense with graphs.

The Spekker Absorptiometer

This instrument was first described by Adam Hilger, Ltd[6] in 1936; although its external form was then somewhat different, the principle was the same as in the familiar H 560 model in general use in metallurgical laboratories to-day. Vaughan[7] advocated the use of the mercury arc as a light source instead of the tungsten lamp with the intention of obtaining monochromatic light; the H 560 model so equipped becomes the H 602. A newer model, the H 760, has recently been introduced (Isbell[8]) which embodies several improvements, but this is at present in less general use.

The principle of the instrument is well known. A single light source illuminates two selenium barrier-layer photocells, which are connected in parallel, in opposition, across a galvanometer. The intensity of light falling on each photocell can be regulated by variable apertures, one of these, on the right-hand side, being calibrated to form the scale of the instrument. The initial setting is made and the readings are taken when the currents produced by the two photocells are equal and the galvanometer deflection, therefore, zero. The extent to which the drum aperture has to be opened or closed is a measure of the difference in density of successive solutions placed in the light beam on the

right-hand side. This design has the great advantage that it makes no assumptions about the linearity of the relationship between current produced and incident light intensity; it is only necessary that this relationship should remain constant over a relatively short period of time, as, provided there is no change in the source intensity, the same current flows at both setting and reading. The double cell design also compensates for small fluctuations in light source intensity, provided the characteristics of the two photocells are similar for the desired wavelengths over a short range.

Light Source

The light source on the Spekker is either a 100-watt tungsten filament projection lamp, or a 125-watt clear tubular "Sieray" mercury vapour lamp, type MB/D, supplied by Messrs. Siemens, Ltd. The former will not under any circumstances give monochromatic light and will, therefore, not be suitable for precise work. The latter, provided suitable filters are used, will give an illumination approaching very closely to monochromatic, and it is assumed throughout this paper that only this source is employed. It is normally supplied by Hilgers for A.C. only and is equipped with a choke, but similar lamps are available from Siemens for D.C. and can be used satisfactorily with a resistance in series. The choke is usually housed in the same cabinet as the instrument, but in view of its vibration we have found it better to place it on a sponge rubber pad on the floor. Individual lamps differ in the steadiness of the arc, and those in which it wanders from one part of the electrode to another should be rejected, as fluctuations due to this cannot be fully compensated by the double cell arrangement. Because of the length of the arc, the collimated beam spreads in a vertical plane and its extremities may be cut off by the fluid meniscus or the bottom of the cell. It is essential to avoid this as it will lead to erratic readings, and if adjustment of the lamp height will not give the beam a clear pathway through the cell and cell-carrier, a fixed diaphragm of say 20 mm. aperture should be placed in the plane of the right-hand (focusing) lens, so that only the central rays are used. As the light is not a point-source, the beam varies in intensity as well as in cross-section, so such a diaphragm will not interfere with the working of the instrument, although it will, of course, alter the calibration and slightly reduce sensitivity.

Compensating Side

The compensating side has two functions, firstly to provide a standard current to which the current on the right-hand (indicating) side can be equated, and secondly to vary the standard with variations in the intensity of the light source. The extent to which the second function compensates for source variations will depend on the similarity of characteristics of the two photocells, which are best matched for the various wavelengths by the makers. There is a water cell on the compensating side, which is intended to protect the photocell from red and infra-red radiation. Nicholas and Pollak[9] have shown, however, that the effect of water cells in this connection is negligible. Moreover the left-hand water cell is very close to the lamp-house and is, therefore, heated considerably (mainly by conduction and convection). This results in the formation of air-bubbles which contribute to drift in the zero setting. Where difference methods are used, there is usually no need for the right-hand water cell either. The setting we recommend for general use on cells not greater than 1 cm. is without either water cell, and is referred to as "air-to-air" setting.*

The compensating aperture can be varied in two ways; the coarse adjustment by an iris, and the fine by rotation of a small oval-section rod. The latter is difficult to use with precision, and it will probably be found easier to use the iris exclusively. It is suggested that the fine adjustment rod be replaced by a fine screw having its point normally at about the centre of the iris; this screw could be inserted through the same hole as the present fine-adjustment.

Drum

The drum consists essentially of a simple collimating lens in the aperture of which is a sliding shutter actuated by an eccentric circular brass cam. The shutter slides in bearings formed by four small screws, and is held against the cam by two small springs, but the possibility of a rocking movement exists, and the return (opening) movement is not sufficiently positive to be fully reliable. For accurate results, therefore, it is necessary not only that the drum should always approach the setting or reading from the same direction, but also that the direction should be the one in which movement is positive—i.e., closing the aperture. It will be found that a smoother movement of the drum can be obtained if the handle supplied is replaced by one about 2 in. long. The scale is graduated in units which approximate to density, but is not individually calibrated; readings cannot, therefore, be substituted for density values in spectrophotometric calculations. For all accurate work, the scale should be re-calibrated, a procedure for which is detailed later. It will also be found worth while to replace the index line which is unnecessarily thick with a finer one. As the instrument will usually be used in a dimly lit room a small spotlight shining on the scale will be found a convenience, and a lens is desirable for setting and reading.

The drum is intended for use by a method different from the one usually adopted. It is calibrated from 0 (at maximum aperture) to ∞ (at zero aperture), and is intended to be set to zero with the unknown in the light path and closed down when the blank is substituted. This involves adjustment of the left-hand aperture, possibly over a wide range, for every reading, so Vaughan[10] simplified the method by introducing the "water-to-water" setting, which, for routine work, need only be made occasionally during a series of readings. For precise work the setting should be checked for every reading, but Vaughan's method still saves time as any variations found will be over a very small range only. This method, of course, results in the scale being used backwards.

Monochromatic Filters

Nicholas and Pollak[9] have shown that with the exception of the 577 mμ line (Calorex + Ilford 606), the filters normally advised and supplied for the isolation of the mercury lines do not, in fact, give anything approaching monochromatic light. The introduction of the monochromatic filters described in the above paper will increase both reproducibility and agreement with Beer's law to a marked extent. In addition, filters will be interchangeable as regards spectral purity. The combinations recommended are given in Table I which is based on the paper quoted above with some

* See section on blanks.

TABLE I.—RECOMMENDED MONOCHROMATIC FILTERS

Wavelength	Combination	Remarks
735 + mμ	Wratten 88A	
" 691 mμ "	Chance ON 15, 2 mm. Ilford 206 Chance ON 13, 2·5 mm.	Transmits 672–709 mμ
" 607 mμ "	Wratten 26 Ilford 804	Includes 607–623 mμ lines
* " Orange-red "	Wratten 25 Ilford 801.	Not monochromatic
* 577 mμ	Ilford 812 Ilford 804	
* 546 mμ	Wratten 16 Wratten 74 Ilford 804	
492 mμ	Ilford 603 (two-component filter) Wratten 5 Ilford 302 Ilford 804 Ilford 803	
* 436 mμ	Chance OB 2, 2 mm. Wratten 2A (treble thickness) Wratten 36	
* 405 mμ	Chance OV 1, 4 mm. Wratten 2B Chance OB 10, 1·5 mm.	
* 365 mμ	Chance OB 2, 1 mm. Wratten 17 Chance OX 1, 3 mm.	

modification necessitated by changes made by the filter manufacturers in their products (see Nicholas & Pollak[35]) If only the Spot galvanometer is available, only those marked with an asterisk can be used. These filters are listed with their components in the order in which light will traverse them ; it will be observed that they include heat-absorbing components, so such filters need not be used in addition.

Gelatine filters are supplied by the makers in any desired size, and to secure uniformity of a number of filters it is suggested that larger sizes be bought and cut up. Cutting is best accomplished with sharp scissors, but the makers' advice to cut through paper should not be followed as this sometimes causes splitting. It will be found most convenient to use a thin piece of card folded so that the top part is the desired size ($1\frac{7}{16}$ in.) and the underneath $\frac{1}{8}$ in. narrower. In cutting, therefore, the upper blade of the scissors will come against the edge of the card but the lower blade will be free. (If both parts of the card jig were the same size the lower would be pushed up by the lower blade of the scissors and tear the filter). Some gelatine filters, especially Ilford 804, will be found to be very brittle. These should be softened by keeping in a constant humidity chamber at a relative humidity of about 90% for a day or so before cutting (a desiccator containing water with an excess of ammonium chloride will suffice). The filter should then be dried over calcium chloride before mounting.

In mounting, it is advisable to use a liberal quantity of the mounting medium, excess of which should be gently squeezed out by hand, taking any air-bubbles with it. Two spring clothes pegs should then be applied to hold the filter together. After two or three weeks, the pegs are removed, surplus mountant is cleaned from the surfaces and the edges are then protected by binding with adhesive cellulose tape. One of the glasses should be marked before mounting as it is in most cases difficult to tell which way round the filter is after it is mounted. As a cement, commercial filter-makers have until recently used Canada balsam. A xylene solution of this will be found quite easy to use, but it has the disadvantages of slow drying and yellowing with age. The latter is undesirable in the violet and ultraviolet regions, so we have tried a number of other media. Polystyrenes (see Lillie, Windle and Zirkle[12]) were abandoned because of poor adhesion to glass and liability to form air-spaces ; but dammar purified according to Bensley and Bensley[13] and dissolved in xylene was satisfactory. We obtained the best results with a solution of 30 g. Piccolyte WW 100 in 15 ml. xylene (see Wicks, Carruthers and Ritchey[14]). This is a synthetic terpene polymer resin produced by the Pennsylvania Industrial Chemical Corporation, Clairton, Penn., which we have found does not yellow appreciably with exposure to light.[15] Unfortunately the manufacturers are not now producing the "WW" (water-white) grades, the nearest available being S 100, which is very slightly yellow. We think it probable that this will be satisfactory, but cannot speak from actual experience.

Glass filters should be optically worked, or a variation in density from one part of the filter to another will be introduced. As such glass varies from melt to melt, it is advisable to check the density at the required wavelength ; suitable values are given by Nicholas and Pollak.[9] Where plain glass is required there is no need for it to be of optical quality and old photographic plates will be found entirely suitable. Some of the compound filters will be nearly 6 mm. thick, so provision should be made on the instrument to take them.

The right-hand filters on the H 560 and H 602 Spekker are placed very close to the lamp house, with the result that they overheat (primarily by conduction and convection, so that the heat-absorbing filter does not in fact give a gelatine filter much protection). In consequence gelatine filters deteriorate, and some show a variation in their transmission with temperature; it is also difficult to change them while hot. Hadley[11] has described a method of securing them on the right-hand side of the drum lens, where it will be found that they show no noticeable rise in temperature. It should be borne in mind, however, that, whether they are in the usual or in Hadley's position, they are approximately in the aperture plane of the drum lens. Different areas of the filters will, therefore, be illuminated at different drum openings, and any defects such as irregularity in thickness of the gelatine, air-bubbles in the mounting medium, or dirt on the surface, enter into every reading. It is, therefore, essential that the same individual filter is always used, and that it is always placed in the instrument the same way round. For more than one wavelength, separate calibrations will be required. Nicholas[16] describes an instrument in which the filter is immediately in front of the photocell. Being here in the image-plane the illuminated area remains constant and even gross irregularities have no effect on readings ; furthermore one calibration suffices for all wavelengths. It should not be unduly difficult to machine a slot in the right-hand photocell housing on the Spekker through which filters could be inserted. It would, of course, be necessary to provide adequate light-trapping, as stray light in this position would be especially detrimental to accuracy.

Recalibration of Drum

If the filters have been transferred to the image-plane, as suggested, a single calibration will suffice for all wavelengths. It is then worth while to construct an entirely new scale, which can be made more open by limiting it to a density range of 0·0–1·0. This can be

done by moving the upper knife-edge, after loosening the two screws securing it in position. The total movement of the lower knife-edge on rotating the drum through 180° is about 11 mm.; shifting the upper one up by 1·2 mm. will, therefore, give the necessary openings for a density range of 1·0, namely, 1·2 mm. (minimum) and 12 mm. (maximum). As most workers will prefer a method of setting as recommended by Vaughan, or as recommended in this paper, to that originally intended, it will be found convenient to label the setting position (∞ on the original scale) 0·0 so that the more rational procedure is adopted of subtracting the blank from the reading instead of the reading from the blank. The use of half the circumference of the drum not only increases the length occupied by one density unit from 146 to 187 mm. but also brings the important setting position to one extreme of the cam movement, with consequent opening out of the scale. If a new scale is being constructed, calibration should be done at the 546 mμ line, which is the only strong single line. Two strips of stainless steel foil* of 0·010 in. thickness are required, with metal blocks soldered at the ends in such a way that they can be held in tension around the drum by screws passing through these blocks. Thin millimetre graph paper is then stuck to one strip so as to form a removable arbitrary scale. Readings on this are related to density by the procedure outlined below. It is then removed, clamped to the bench alongside the other strip, and the final scale drawn on the latter with scriber and square.

If, on the other hand, filters are in an aperture-plane (whichever side of the drum), separate calibrations will be required for each individual filter, and it is important that filters should always be used the same way up. It is then hardly worth while to do more than construct a correction graph (or, preferably, table).

The procedure for recalibration is as follows. An optically worked glass neutral filter (e.g., Chance ON 31 or ON 32) will be required, of such thickness as to have a density of 0·4–0·5 at 546 mμ; a convenient size is 1 × 1½ in. It should be calibrated (e.g., by the National Physical Laboratory) for the desired wavelengths; it is as well to allow two months or so to elapse between working and calibrating so that self-blooming of the surface ceases. A filter-holder should be constructed to hold it in the cell-carrier in a plane accurately normal to the optical axis. Another filter of lower density (which need not be known precisely) will also be needed; an empty glass cell can be used for this. With the required monochromatic filters in place, the instrument is set air-to-air at the required starting position (1·0 if a correction graph is being constructed, or a position on the arbitrary scale corresponding to ∞ on the drum for a complete new calibration). A reading of the standard filter is made (referred to as "Standard 1 ×"); without moving the drum, the neutral filter is then removed and the instrument reset by means of the zero adjustment. A second reading of the filter is then taken ("Standard 2 ×"). We have now two points on the scale corresponding to known densities. The Spekker is again set to the starting position, and a similar procedure adopted with the empty cell. By this means the whole scale is mapped out in equal, but so far unknown, increments of density (actually about 0·07). All these readings should be made in duplicate, and means of consistent series used for final calculations. By interpolation, it is then possible to express the two known density values (Standard 1 × and Standard 2 ×) in terms of the density of the glass cell. The value of the latter is calculated (to five places of decimals to avoid cumulative error later). Scale readings can then be plotted against density, and additional points should be obtained, on any part of the graph showing considerable curvature, by starting from suitable drum readings on a straighter portion.

Table II is an example of drum calibration at 546 mμ on the Spekker available to us. This was for the purpose of constructing a correction graph, and the density values are, therefore, related to readings on the original scale starting at 1·0. It will be observed that two separate values are calculated for the density of the glass cell, one for the range 0·0–0·488 and the other for 0·488 to the end of the scale. They only differ in the fourth place of decimals (by 0·0003) probably owing to the use of different areas of the cell at different drum openings. To avoid cumulative error, the two different values were used in constructing the corresponding ranges of the correction graph.

The standard neutral filter will also be found very useful for periodic checking of the instrument; readings "Standard 1 ×" and "Standard 2 ×" should agree with expected values to within ± 0·001. It should be noted that determinations of density by the National Physical Laboratory are subject to a possible error of ± 0·5%.* It is, therefore, not possible to obtain *absolute* values of density to a greater accuracy than this if a single N.P.L. calibration is relied on. This need not, however, affect the reproducibility of results on one instrument, or the comparability of results on different instruments, provided they are all calibrated with the aid of the same standard filter.

Optical Glass Cells

The cells normally used with the Spekker are the three-piece fused U-bend type supplied by The Tintometer, Ltd. The suppliers [17] state that all cells above 1 cm. optical depth are correct to within ± 1% in the direction of the optical path; smaller sizes naturally show greater relative variations. In addition, deviation from parallelism of the optical faces is sometimes found. As the clear glass plates forming the faces of the cell are plane-parallel and are not worked after fusing†users are recommended to check the overall width of the cells with a micrometer at various places in the area used as an optical path,‡ and to reject any cell which does not show substantial uniformity of optical depth. In the case of cells of 2 cm. and larger, the thickness of the glass can also be measured directly with a micrometer and the optical depth determined by subtraction. Smaller cells can be calibrated by measuring the density of the same coloured solution in a known accurate cell and in the unknown cells; Table III illustrates this method applied to cell standardisation. The 546 mμ line was used, with solutions of cobalt sulphate. A solution of 200 g./litre of $CoSO_4 \cdot 7H_2O$ (B.D.H., ordinary quality) was prepared. The salt was not dried beforehand, and its purity was not known; the value of

* Brass foil can be substituted, provided care is taken not to elongate it during clamping. It will require lacquering after the scale is drawn.

* Personal communication.
† Tintometer, Ltd., personal communication.
‡ The manufacturers do not claim accuracy for that part of the cell more than 30 mm. below the top edge.

$E^{1\%}_{1cm.}$ obtained, therefore, applies only to this particular sample, but this does not affect the accuracy of the measurements of cell depth made. Solutions of other concentrations were prepared by dilution of the original solution, and all measurements were made on the same day. The Nicholas[16] absorptiometer was actually used, but the method is equally applicable to the Spekker. The standard cell was one specially selected by The Tintometer, Ltd., and found by them to have an optical depth of 10·000 ± 0·005 mm. The calibration of all available cells for optical depth renders unnecessary the usual procedure of preparing separate standardisation graphs for different sizes of cell, provided Beer's law is followed.

Blanks

Space is provided on the right-hand side of the Spekker for three solution cells, and it is usual to use two, one for the unknown solution, and one for water or other blank. For routine work, the cells are assumed to be similar, and the initial setting is made with the blank in position; its value is then automatically allowed for in reading the unknown. For precise work the assumption of the similarity of the cells cannot be made, as not only will they be of slightly different optical depths, but also clouding of the inner surfaces may cause a difference in density between the two cells, and in the same cell from day to day. The blank must, therefore, be read in the same cell as the unknown, and on the same day. If this is done, it does not matter a great deal whether the initial setting is "air-to-air" or "air-to-water." The former is our usual practice with short cells, but the latter would appear to present advantages with cells of 2 cm. or more, or for work of the utmost precision with any cell, in ensuring that optical conditions remain identical at setting and reading.

Blanks will usually be of small value, and if read from such a setting as 1·0,* it will be necessary to read them by interpolation in an inaccurate region of the scale. It is, therefore, advisable to read them from a much lower setting. It should be remembered that on the Spekker, as supplied, the relationship between drum readings and density may not be exactly linear; it is, therefore, meaningless to add or subtract readings unless they have been converted to density beforehand.

Photocells

The photocells used on the Spekker are the selenium on iron barrier-layer type supplied by Messrs. Evans Electroselenium, Ltd., and their characteristics are described in the maker's booklet.[18] In the design of the Spekker, no assumption as to the linearity of the response is involved, and since the area and position of the illuminated region of the right-hand photocell remain the same throughout, uniformity of response from one part of the photocell to another is unimportant. Messrs. Hilger[19] state that it is advisable to make readings as soon as possible after the photocell is illuminated to avoid the effect of fatigue. This is, however, directly contrary to the advice given by the photocell makers for their colorimeter[20] and by Clayson and Scott[21] and Bell and Guthmann[22] for theirs.† Provided the two photocells are well matched, it will not be found that fatigue produces any appreciable effect, but differential fatigue may be more noticeable in the red and infra-red. It will then be found that the zero drift is fairly rapid immediately the shutter is opened, but settles down to a lower, steady value or disappears altogether after a short while. If drift from differential fatigue is experienced, it is advisable to take readings after the photocells have been illuminated long enough to reach a steady state. It should be noted that there is one important non-electrical cause of zero drift in the instrument as generally operated—namely, the formation of air bubbles in the left-hand water cell as the result of heating.

TABLE II.—EXAMPLE OF CALIBRATION OF SPEKKER DRUM AT 546 mμ

	Mean reading	Density (known)	Density (calculated)	Density (approximated)
Standard 1 ×	0·543	0·488	—	—
Standard 2 ×	0·062	0·976	—	—
Glass cell 7 ×	0·5685	—	—	—
Glass cell 8 ×	0·509	—	—	—
Glass cell 7·4286 × (by linear interpolation)	0·543	0·488	—	—
Glass cell 1 × (up to density 0·488)	—	—	0·06569	—
Glass cell 14 ×	0·128	—	—	—
Glass cell 15 ×	0·048	—	—	—
Glass cell 14·825 (by linear interpolation)	0·062	0·976	—	—
Glass cell 7·3961 × (by subtraction)	—	0·488	—	—
Glass cell 1 × (density 0·488–0·976)	—	—	0·06598	—
Glass cell 1 ×	0·9375	—	0·06569	0·066
Glass cell 2 ×	0·875	—	0·13138	0·131
Glass cell 3 ×	0·8115	—	0·19707	0·197
Glass cell 4 ×	0·7515	—	0·26376	0·264
Glass cell 5 ×	0·691	—	0·32845	0·328
Glass cell 6 ×	0·631	—	0·39414	0·394
Glass cell 7 ×	0·5685	—	0·45983	0·460
Glass cell 8 ×	0·509	—	0·52552	0·526
Glass cell 9 ×	0·4455	—	0·59150	0·592
Glass cell 10 ×	0·383	—	0·65748	0·657
Glass cell 11 ×	0·323	—	0·72346	0·723
Glass cell 12 ×	0·262	—	0·78944	0·789
Glass cell 13 ×	0·1975	—	0·85542	0·855
Glass cell 14 ×	0·128	—	0·92140	0·921
Glass cell 15 ×	0·048	—	0·98738	0·987

Galvanometer

The Spekker equipped with the Unipivot pointer galvanometer may now be considered as obsolete, as the

TABLE III.—CALIBRATION OF CELLS

Cell No.	Concentration g./litre	Intrinsic Density	$E^{1\%}_{1cm.}$	Optical Depth cm.
Standard ..	60	0·523	0·087167	1·0000 ± 0·0005 (known)
	80	0·697	0·087125	—
	100	0·872	0·087200	—
			0·087165 (mean)	
1	60	0·522	—	0·9981
..	80	0·697	—	1·0000
	100	0·871	—	0·9988
				mean 0·9990
3	60	0·523	—	1·0000
	80	0·699	—	1·0024
	100	0·874	—	1·0023
				mean 1·0016
5	20	0·350	—	2·0078
	40	0·701	—	2·0107
	50	0·875	—	2·0078
				mean* 2·0088
11	80	0·340	—	0·4876
	160	0·679	—	0·4869
	200	0·849	—	0·4870
				mean 0·4872

* This refers to the original calibration of the drum. If the full process of calibration described earlier has been applied, blanks can be read satisfactorily from the usual setting.

† All these use the same photocells.

* Direct measurement of cell No. 5 with a screw micrometer in the area used as optical path gave a mean optical depth of 2·0062 cm., with a maximum deviation of ± 3$0\mu$. The optical depth at the centre of this area was identical with the mean.

TABLE IV.—MOLECULAR EXTINCTION COEFFICIENT OF POTASSIUM CHROMATE

Concentration (mol./litre)	Mean Density	Density Minus Blank	Molecular Extinction Coefficient at 436 mμ
$1{\cdot}2356 \times 10^{-3}$	0·4280	0·3850	311·55
$1{\cdot}8534 \times 10^{-3}$	0·6221	0·5791	312·46
$2{\cdot}4712 \times 10^{-3}$	0·8134	0·7704	311·76
nil (blank)	0·0430	nil	—
		mean	311·92
		mean corrected to 20° C.	312·29

instrument is now supplied with the Cambridge Spot giving a deflection of 170 mm./μA. This is satisfactory for many purposes, but if it is proposed to measure high densities or use the weaker lines, a greater sensitivity is necessary. We have found a Tinsley galvanometer No. VS6/45 giving up to 1,950 mm./μA (variable by a rotary switch) suitable for most purposes, although very occasionally the need is felt for still greater sensitivity. The Spekker has a mercury switch in series with the galvanometer which occasionally gives trouble; it is inconvenient in use as it has to be depressed for every reading, and to avoid this a foot switch has been described. If, however, the instrument is used with even reasonable care, no currents will normally be sent through the galvanometer greater than would cause a full-scale deflection; it is, therefore, suggested that the mercury switch be removed and replaced by a tumbler or knife switch in *parallel* with the galvanometer. This would be left open throughout a series of readings, but closed when the instrument is not in use, thus affording the galvanometer protection against mechanical shocks which a series switch cannot give. It will often be found that touching the instrument causes a galvanometer deflection due to part of the photocell circuit passing through the main casting. To avoid this, and also to protect the galvanometer from a possible short circuit from the lamp supply, it is essential that the base of the Spekker should be adequately earthed.

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It is not usual to take any special precautions to control temperature in photoelectric absorptiometry, although it can interefere in two ways. It can act directly on a coloured substance, as, for example, by altering the equilibrium position of a reaction. Stross[23] in his Titan yellow method for magnesium in aluminium alloys finds under his conditions the exceptionally large temperature coefficient of extinction of $+0{\cdot}016$/° C. Davis and Bacon[24] find a negative temperature coefficient with ferric thiocyanate ($-0{\cdot}006$/° C. under their conditions) and state that to get results reproducible to ± 1% it is necessary to control the temperature to within ± 2° C. This type of temperature effect is peculiar to the individual substance, and so will not be considered further in this account, which is intended to be of a general nature. The effect of expansion, however, enters into all determinations in greater or less degree: expansion of the solution produces a fall in concentration (w/v), and of the cell an increase in thickness. The cubical coefficient of expansion of water at 20° C. is $2{\cdot}07 \times 10^{-4}$, and the linear coefficient of the glass cell is $8{\cdot}5 \times 10^{-6}$ (Tintometer, Ltd.[25]) If at 20° C. $E_{20} = kcl$ then at 21° C. $E_{21} = k \times (c/1{\cdot}000207) \times 1{\cdot}0000085l = 0{\cdot}9998\ kcl$

Therefore, $E_{20} - E_{21} = 0{\cdot}0002\ kcl$

It will be seen, therefore, that a temperature differen of 5° C. is required to produce a discrepancy of 0·1 when working with aqueous solutions. Sulphuric ac solutions, however, have a higher coefficient, while th of organic solvents is still greater: the latter especiall may require close temperature control.

Accuracy in Normal Use

As an example of the accuracy to be expected in normal use, we give a determination of the molecular extinction of potassium chromate, which is known to obey Beer's law strictly.[26] Analar potassium chromate, without further purification, was dried at 100° C. and dissolved in 0·1 N potassium hydroxide (to prevent formation of dichromate). Various concentrations were used, and the molecular extinction was calculated from density readings at 436 mμ, 0·1 N potassium hydroxide being used as blank. The temperature of the solutions was 26·1 ± 0·9° C. Table IV shows the results and the densities given are the means of sets of eight readings, individual readings differing from the mean of the respective set by not more than 0·25%. The mean molecular extinction coefficient derived from the 24 single determinations is 311·92, with a coefficient of variation of 0·16%; no single value differed from the grand mean by more than 0·37%. Corrected for temperature, the extinction becomes 312·29; this should be compared with the result of von Halban and Siedentopf[27], who, using a Zeiss monochromator and a photoelectric null method, obtained a mean value of 313·8 with a maximum deviation of single values from the mean of 0·5%. The discrepancy between our value and theirs is 0·5%; factors contributing to this are the possible error of ± 0·5% in our primary standard of density and, possibly, the lack of temperature control by von Halban and Siedentopf. Our result should also be compared with the transmission of 0·861 for a 0·0400 g./l solution in a depth of 10·00 mm. determined by Haupt[28], which corresponds to a molecular extinction of 315·6, being 1·05% greater than our result. It should be noted that in Haupt's figures there is said to be "considerable uncertainty in the third decimal" and it would only require a difference of 0·0014 in transmission to make her result identical with ours.

Measurement of High Densities

The useful density scale will extend up to about 1·0, and cases will arise where a solution under test is too dense to give a reading. Obviously it could still be measured if a smaller cell were available or if it could be diluted. The latter course is, however, only permissible where the specific extinction is unaffected by changes in the concentration of reagents. A very simple way of extending the scale is by the use of neutral filters. The density of these is determined on the Spekker at the wavelength required, and the instrument then set with a neutral filter on the right-hand side (i.e., air-to-neutral-filter). The unknown is then substituted for the filter, and its density is given by the measured density plus that of the filter. High[29] used a similar technique of setting with a filter on the right-hand side; his note gives little detail, but he appears to have worked by calibrating with the filter *in situ* instead of by adding densities. Vaughan[10] and Stross[30] have mentioned a technique with neutral filters, but they use them on the left-hand side, adding them after the absorptiometer has

been set. This is very convenient, but defeats one of the principles of the design of the instrument, namely, that the light intensity on the indicating photocell should be the same at setting and reading. For precise work such filters should only be used on the right-hand side. An alternative method is to measure a dense solution at another wavelength where its extinction is lower; an example of this is given later.

Estimation of More than One Unknown

For this purpose it is necessary to know the characteristic absorption curve of the various coloured compounds met in analysis. Where reliable data are not available, these can be constructed by measuring the solutions at all available wavelengths, and plotting log d against wavelength. Such a curve has a shape characteristic of the coloured compound or ion under investigation, and so long as Beer's law is followed this shape is independent of concentration, change in which only affects the position of the whole curve. More than one constituent will usually be present in metallurgical analysis; the characteristic curves should, therefore, be superimposed to ascertain the wavelengths where negligible interference occurs.

We quote as an example the case of manganese and chromium in steel. From the characteristic curves it is obvious that the Cr_2O_7 ion will not interfere at a wavelength of 546 mμ or higher. Manganese can, therefore, be determined in the presence of chromium at these wavelengths without the use of a correction factor. From the shape of the absorption curve of the MnO_4 ion it can be seen that the 546 mμ filter is eminently suitable for measuring low concentrations, while the 577 mμ filter will serve well at medium concentrations. Still higher manganese contents can be determined with the "607 mμ" filter (or the "orange-red" when only the Spot galvanometer is available). In this method of analysis, the instrument was first calibrated separately for manganese and then for chromium. The concentration during oxidation was 1·75 N sulphuric acid; 0·94 N phosphoric acid; 2·8 g./l silver nitrate; and 29·4 g./l ammonium persulphate. The final concentration was: artificial alloy (iron + manganese and/or chromium) 2·5 g./l; 1·49 N sulphuric acid; 0·8 N phosphoric acid; 2·4 g./l silver nitrate; and 2 g./l urea. Difference methods were used throughout. The blank for manganese was obtained by the addition of 0·05 ml. of 10% sodium nitrite to the cell followed by stirring, while the blank for chromium was made by adjusting the concentration and acidity of the original solution to that of the test without oxidising. Manganese was read with 577 or 546 mμ filters, and chromium with 436 mμ. In preference to drawing calibration graphs a conversion factor was calculated. Table V shows the process of calibration.

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Metal	Filters	Cell	% Metal	Density Difference Between Test and Blank	Conversion Factor
Manganese	577 mμ	1 cm.	0·60	0·218	2·754
			1·20	0·437	2·746
			1·80	0·653	2·757
			2·40	0·874	2·743
				mean	2·750
				coefficient of variation	0·238%
Manganese	546 mμ	1 cm.	0·24	0·256	0·9376
			0·48	0·514	0·9337
			0·72	0·770	0·9350
				mean	0·9354
				coefficient of variation	0·212%
Chromium	436 mμ	2 cm.	1·20	0·162	7·408
			2·40	0·324	7·408
			3·60	0·490	7·347
			4·80	0·652	7·362
			6·00	0·814	7·369
				mean	7·379
				coefficient of variation	0·376%

TABLE VI.—ESTIMATION OF MANGANESE AND CHROMIUM IN SAME SOLUTION

Solution	Mn Taken	Cr Taken	Difference of Densities 577 mμ	546 mμ	436 mμ	Mn Found	Cr Found
	%	%				%	%
(a)	1·20	3·60	0·439		0·489	1·207	3·608
(b)	2·40	1·20	0·874		0·163	2·403	1·203
(c)	0·60	3·60	0·218		0·486	0·600	3·586
(d)	0·60	3·60		0·642	0·486	0·601	3·586

Artificial steel solutions were then prepared containing both manganese and chromium, and Table VI shows the results of analyses by the above method. It will be seen that their accuracy compares favourably with those of Lingane and Collat[31] obtained on a Beckman spectrophotometer.

The above method is simplified by the fact that it has been possible to eliminate completely the effect of unwanted ions at each wavelength. If this had not been possible, it would still be practicable to make the estimation by readings at two wavelengths, provided wavelengths could be found at which the extinctions of the two ions were sufficiently different. The calculation of the result would, however, be complicated by the need to solve simultaneous equations.

It has been mentioned earlier that conversion factors are more convenient than calibration curves; it will be found that they are also more accurate. The usual calibration graph on millimetre paper requires interpolation to 0·1 or at most 0·2 mm. to use the full accuracy of the instrument, and errors in reading the graph may therefore be introduced. Factors will normally be calculated for each concentration used, and their agreement will be a more conclusive demonstration of conformity with Beer's law than the apparent linearity of a small-scale graph. The low coefficients of variation in Table V should be noted.

Relationship Between Accuracy and Concentration

The effect of small errors in an analytical method can usually be minimised by increasing the sample size so that the relative error is negligible. In photoelectric absorptiometry another factor must, however, be considered, namely, the greater absolute accuracy with which small densities can be read. In an instrument with a purely electrical calibration, Twyman and Allsopp[32] have shown mathematically that a compromise between the two conditions is necessary, giving a maximum relative accuracy at a density of 0·4343 with sufficiently accurate results between 0·2 and 0·8. The relationship between increases in current or light intensity and in density (dI/dd) is not however the sole factor at work on the Spekker. The above authors state that here the greatest accuracy is between 0·6 and 1·0. Mechanical factors in the design of the instrument will largely influence this.

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				mean	0·9354
				coefficient of variation	0·212%
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			4·80	0·652	7·362
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(b)	2·40	1·20	0·874		0·163	2·403	1·203
(c)	0·60	3·60	0·218		0·486	0·600	3·586
(d)	0·60	3·60		0·642	0·486	0·601	3·586

Artificial steel solutions were then prepared containing both manganese and chromium, and Table VI shows the results of analyses by the above method. It will be seen that their accuracy compares favourably with those of Lingane and Collat[31] obtained on a Beckman spectrophotometer.

The above method is simplified by the fact that it has been possible to eliminate completely the effect of unwanted ions at each wavelength. If this had not been possible, it would still be practicable to make the estimation by readings at two wavelengths, provided wavelengths could be found at which the extinctions of the two ions were sufficiently different. The calculation of the result would, however, be complicated by the need to solve simultaneous equations.

It has been mentioned earlier that conversion factors are more convenient than calibration curves; it will be found that they are also more accurate. The usual calibration graph on millimetre paper requires interpolation to 0·1 or at most 0·2 mm. to use the full accuracy of the instrument, and errors in reading the graph may therefore be introduced. Factors will normally be calculated for each concentration used, and their agreement will be a more conclusive demonstration of conformity with Beer's law than the apparent linearity of a small-scale graph. The low coefficients of variation in Table V should be noted.

Relationship Between Accuracy and Concentration

The effect of small errors in an analytical method can usually be minimised by increasing the sample size so that the relative error is negligible. In photoelectric absorptiometry another factor must, however, be considered, namely, the greater absolute accuracy with which small densities can be read. In an instrument with a purely electrical calibration, Twyman and Allsopp[32] have shown mathematically that a compromise between the two conditions is necessary, giving a maximum relative accuracy at a density of 0·4343 with sufficiently accurate results between 0·2 and 0·8. The relationship between increases in current or light intensity and in density (dI/dd) is not however the sole factor at work on the Spekker. The above authors state that here the greatest accuracy is between 0·6 and 1·0. Mechanical factors in the design of the instrument will largely influence this.

It should be remembered that in normal use the

Spekker reading is a measure not of a density but of a difference in density between the right-hand water cell and the solution cell. It is obvious that, provided a sufficiently open part of the scale is used, this difference can be measured with greater absolute accuracy the closer it approaches zero. If, then, the water cell contains not water or a blank but a standard solution approximating in concentration to the unknown, the difference in concentration between standard and unknown can be read with high absolute accuracy whatever the density of the two solutions. If this density is made high the relative accuracy will also become high. This procedure has been described at some length, largely from a mathematical standpoint, by Hiskey[33] and has been applied to the Beckman spectrophotometer by Bastian *et al.*,[34] and has been given the name of differential spectrophotometry. The limiting factor in the increase of accuracy by this method is the sensitivity of the galvanometer, as it is necessary that, with the standard solution in position, a full scale deflection should be obtainable at the zero setting. Of the filters described by Nicholas and Pollak that of lowest density (546 mμ) gives full sensitivity on the Spekker at an air-to-air setting of 1·52 (on the unmodified scale). This means that, even by the differential method, densities greater than about 1·52 cannot be measured with accuracy. Replacing the galvanometer by a more sensitive one will, of course, enable higher densities to be used, but the sensitivity required varies with the antilogarithm of the density, and therefore rises sharply with increase in the latter. As an example of what could be achieved by this method, let us assume the instrument set air-to-standard 0·4 (on the unmodified scale) with a standard of density 2·5. The unknown gives a reading of 0·391, and, because of the region on the drum and the closeness of the two values, we can rely on this difference of 0·009 to ± 0·001 or better. It will be seen, therefore, that we are measuring a density of 2·5 with an error of ± 0·001, i.e., a relative error of ± 0·04%. It will be obvious that it will be necessary to check the zero with standard solution in both cells. It will also be obvious that in this technique the standard in the water cell is primarily fulfilling the function of an added density, and could be replaced by a neutral filter of density close to that of the unknown solution, provided the high range of concentration is calibrated by standard solutions. The method would then appear to be similar to that adopted by High,[29] although the latter author gives no figures by which the accuracy attainable can be judged.

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The Polarographic Determination of Chromium in Certain Aluminium Alloys

continued from page 330

chromium. Then continue in the same manner as for the sample, recording the polarogram at the same sensitivity. The chromium solution should be carefully prepared from AR potassium chromate. The strength of the solution may be confirmed volumetrically.

TABLE II.—TYPICAL RESULTS OF THE METHOD

Sample Identification	Alloy Type	Polarographic % Cr	Volumetric % Cr
ML 58	Binary Al-Cr	0·152	0·153
ML 59	"	0·303	0·309
STP 1	HID 33	0·248	0·255
SP 473	HID 44	0·099	0·093
SP 474	"	0·212	0·206
SP 475	"	0·418	0·412
65 ST	"	0·234	0·223
SP 469	HID 22	0·107	0·110
SP 470	"	0·208	0·207
SP 471	"	0·394	0·396
SP 472	"	0·399	0·405
NL 77	HID RR77	0·115	0·108
X 1	"	0·142	0·141
SP 512	"	0·147	0·146
X 7	"	0·233	0·240
X 10	"	0·253	0·255
X 8	"	0·401	0·407

As an alternative to the above, alloys of accurately known chromium content may be used. It is advisable to use the modified procedure with electrolysis on both standards and samples when the greater amounts (more than 0·25%) of copper are known to be present.

The concentration of alloying elements shown in Table I and lead, tin and antimony in the small percentages normally encountered in these alloys do not appear to interfere with the method.

So far, the majority of the chromium contents investigated lie between 0·1 and 0·5%. However, preliminary tests indicate that the determination of lower amounts is practicable and the procedure can probably be used for chromium contents in the range 0·01–0·1% where volumetric methods are less convenient.

Further work is in progress to extend the above method to other alloys.

Acknowledgment

The authors wish to thank the Directors of Messrs. High Duty Alloys, Ltd., in whose research laboratories the method was developed, for permission to publish this article.

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The Polarographic Determination of Chromium in Certain Aluminium Alloys

By E. C. Mills, A.R.I.C. and S. E. Hermon

Research Laboratories, High Duty Alloys, Ltd.

A semi-micro method for the determination of chromium in binary chromium-aluminium and certain aluminium alloys is described. When copper is present in amounts greater than about 0·25% a modified procedure is recommended. Normal constituents of the alloys tested do not interfere, nor do minor amounts of the elements more rarely present. The method has been used for chromium contents covering the range 0·1–0·5%, but this range could probably be extended.

CHROMIUM is a normal constituent of a number of aluminium alloys, and it was considered that a polarographic method for this element would provide a simple alternative to the normal photometric and volumetric methods, particularly where only small quantities of sample are available. During the polarographic determination of zinc in some of these alloys, using an alkaline supporting electrolyte, it was noticed that the chromate ion gave a well-defined wave ($E_{\frac{1}{2}} = -0{\cdot}85$ v. versus S.C.E.* in N.NaOH)[1] immediately below that of zinc ($E_{\frac{1}{2}} = -1{\cdot}50$ v. versus S.C.E. in N.NaOH).[1] Reference to literature showed that Lingane and Kolthoff[2] obtained a well-defined wave in 0·1–1 N sodium hydroxide as supporting electrolyte; the half-wave potential being −0·85 v. versus S.C.E. They also found that the diffusion current was proportional to the concentration of chromate ion.

In addition, procedures using an alkaline supporting electrolyte (1–2 N) for the polarographic determination of chromium in steel and other iron products have been described by Stackelberg *et al*[3] and Thaneiser and Willems.[4]

Below are summarised the series of experiments leading up to the authors' final method, with particular reference to certain aluminium alloys and binary chromium-aluminium, the composition ranges of which are given in Table I.

Preliminary Experimental Work

Thaneiser and Willems[4] found that the diffusion current of the chromate ion decreased markedly when the sodium hydroxide concentration was increased above 1 N. As this concentration was likely to be exceeded in the final solutions derived from the processing of aluminium alloys, the magnitude of this effect was studied with standard chromate solutions in the absence of aluminium or the other constituents likely to be present in alloys. The tests covered the equivalent of 0·4–0·5% chromium on the final sample weight, and sodium hydroxide concentrations of 0·2–3·2 N. Deviations from the wave height recorded in a 1 N solution—expressed as percentage change—were plotted against sodium hydroxide concentration, as shown in Fig. 1. Later, in similar experiments, where the sodium hydroxide was partially neutralised by an addition of sulphuric acid, the resulting wave height was not proportional to the actual "free" sodium hydroxide content as indicated in Fig. 1, but was somewhat lower in magnitude.

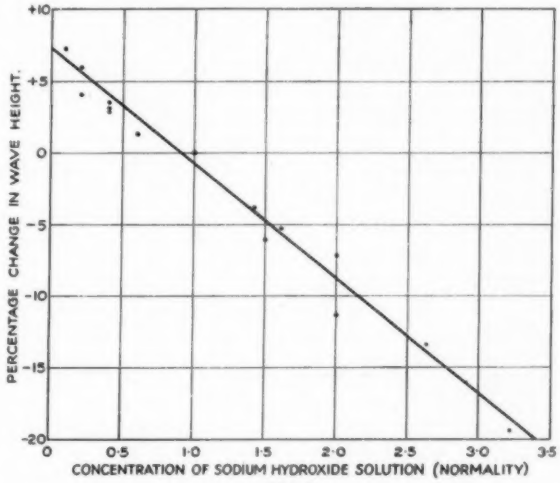

Fig 1

The addition of a small amount of suppressing agent was found to be necessary to prevent appearance of maxima. Fig. 2 shows typical results of the addition of varying amounts of gelatin (up to 0·044% w/v gelatin in the final solution) on the chromium wave height—expressed as percentage change—with reference to a solution containing 0·2 ml. of 0·55% gelatin in the final 25 ml. bulk. It was noted that different sources of gelatin gave slightly different curves. The tests were carried out in solution 1 N with respect to sodium hydroxide, and with the equivalent of 0·4–0·5% chromium present

TABLE I.—COMPOSITION OF ALLOYS ANALYSED

Alloy	D.T.D. Specification	Cu %	Ni %	Mg %	Fe %	Si %	Ti %	Mn %	Zn %
HID 22	310C, 440 606, 634	Up to 0·15	—	1·0–2·75	0·2–0·75	0·2–0·6	Up to 0·2	0·1–0·5	Up to 0·1
HID 33	180B	Up to 0·10	—	3·0–3·5	Up to 0·4	Up to 0·4	—	Up to 0·2	—
HID 44	346	Up to 0·15	Up to 0·2	0·4–1·7	0·2–0·6	0·3–1·3	Up to 0·15	0·25 0·75	Up to 0·1
HID RR77	363A 683, 687, 693	Up to 1·9	—	2·0–2·5	Up to 0·3	Up to 0·3	—	Up to 0·3	5·0–7·0
Binary Chromium Aluminium	—	—	—	—	Up to 0·2	Up to 0·2	—	Up to 0·05	—

* Standard Calomel Electrode.

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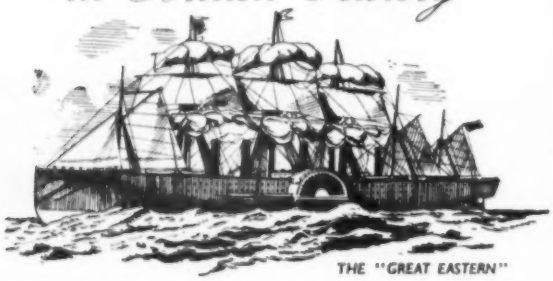

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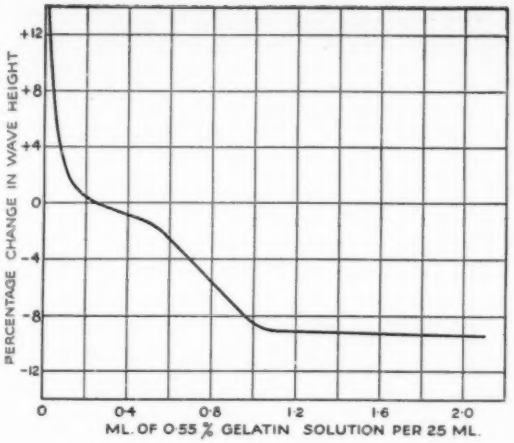


Fig. 2

as chromate as before. Variations of chromium over the range 0·1–1·0%, using sensitivities from 1/7 to 1/100, showed that the wave height increased almost linearly with the concentration of chromium.

In the earlier experiments, deoxygenation was effected by bubbling nitrogen through the solution in the polarographic cell, but it was found later that equally satisfactory results could be conveniently obtained by the use of sodium sulphite (in an alkaline solution) as reported by Kolthoff and Laitinen.[5]

Tests carried out with chromium in the presence of aluminium, gave somewhat smaller wave-heights than those resulting from the chromium solutions alone. Additions of up to 7% zinc and 2·5% manganese did not further affect the wave height.

Summary of Techniques Investigated for the Analysis of Alloys

Initially, solution of the alloys was attempted in sodium hydroxide, with additions of hydrogen peroxide, to obtain the chromium as sodium chromate. The resulting precipitate was filtered or centrifuged off, and then dissolved in sulphuric acid with hydrogen peroxide addition, the solution neutralised with sodium hydroxide in the presence of hydrogen peroxide or sodium peroxide, re-filtered and the two filtrates combined. Aliquots of the combined filtrates, after decomposition of excess hydrogen peroxide by boiling, were deoxygenated and the chromium determined polarographically.

It was discovered that large amounts of chromium were retained by the precipitates. The amount retained could not be correlated with the presence of any one or more of the normal constituents of the alloys. It did, however, in most cases, appear to increase with increasing magnesium and chromium contents.

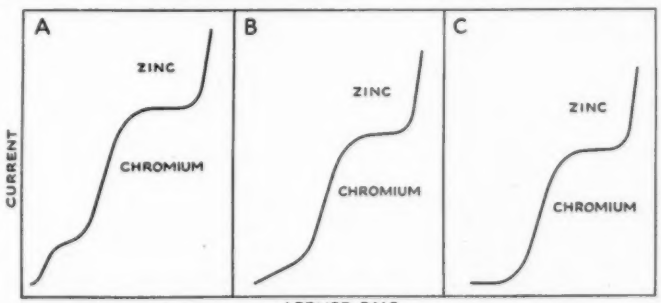
Therefore, as an alternative, the initial dissolution of the alloys was effected in acid and this acid solution was then neutralised by pouring into sodium hydroxide solution containing hydrogen peroxide. The solution was then boiled to decompose the excess peroxide, evaporated down and the precipitate centrifuged or filtered off. An aliquot of the solution was then deoxygenated and the chromium determined by the polarograph. Recovery of chromium was again incomplete but to a much lesser degree than previously. It was later found preferable to filter off the precipitated hydroxides at an early stage to minimise solution of the copper (particularly in alloys containing more than about 0·3% copper) which had an effect on the residual current, causing distortion of the bottom plateau of the chromate wave. (Graph A, Fig. 3).

The next step was essentially the same as the above, except that the chromium in the acid solution was oxidised with sodium persulphate before pouring into sodium hydroxide containing hydrogen peroxide. Potassium and ammonium persulphates were also tried as alternatives, but the sodium salt was found to be most satisfactory. Slightly low results were still obtained and, as previously, an appreciable amount of the copper present passed into the filtrate. The loss of chromium appeared particularly noticeable in the presence of high magnesium contents (more than 2% Mg.).

The hydrogen peroxide was then omitted entirely whereupon good recoveries of chromium were obtained, and the effect of copper on the wave form was considerably reduced. For all practical purposes, the copper interference was now negligible in alloys with copper contents of less than 0·25%; however, in alloys of greater copper content the interference was still noticeable, particularly when low chromium contents (less than 0·25%) were being determined, using high sensitivities. In earlier experiments, variation of the final alkalinities between 1·0 and 2·3 N with respect to sodium hydroxide, and additions of sodium carbonate up to 0·5 g., were unsuccessful in preventing solution of the copper.

Eventually the difficulty was readily solved by electrolysis of the copper after a modified initial solution procedure. Spiral, sheet and cylindrical gauze platinum cathodes were tried in turn, the last-named being found to be most satisfactory.

Graphs B and C, Fig. 3 show the distinct improvement in wave form, with and without electrolytic removal of copper, using the two final procedures. The equivalent of 0·444% chromium on a 250 mg. sample was added to each of a series of chromium-free alloys and, as electrolytic removal of the copper was not usually found essential when determining this amount of chromium in the presence of up to 1% copper, the simpler technique was used for all the alloys. Using a sensitivity of 1/30,



A. RR 77 (1%Cu), 0·44%Cr, SENSITIVITY $\frac{1}{30}$–PRELIMINARY TECHNIQUES.
B. RR 77 (1%Cu), 0·144%Cr, SENSITIVITY $\frac{1}{8}$–FINAL TECHNIQUE–NO ELECTROLYSIS.
C. RR 77 (1%Cu), 0·144%Cr, SENSITIVITY $\frac{1}{8}$–MODIFIED TECHNIQUE–WITH ELECTROLYSIS.

Fig. 3

[5] Kolthoff, I. M. and Laitinen, H. A., *Science*, **92**, 152 (1940).

the relative heights of the waves observed which show a comparatively small variation, are as follows :—

Alloy Type	Wave Height
Binary Chromium-Aluminium ..	62½
HID RR77 (1% Cu)	64½
HID RR77 (0·4% Cu)	64½
HID 22	62½
HID 44	63½

It was also noticed that the factors obtained for the percentage of chromium which is represented by unit height division and referred to a common sensitivity, e.g., 1/10, increase slightly with increasing amounts of chromium. One series of experiments gave the following height variations for additions of chromium to a HID 22 type alloy.

% Chromium	Sensitivity	Wave Height	Factor
0·16	1/10	60½	0·002656
0·24	1/15	60	0·002667
0·32	1/20	58½	0·002735

This effect is probably due to slight retention of the chromium in the hydroxide precipitate.

The Final Method

All reagents should be of "Analytical Reagent" quality and solutions should be freshly prepared where necessary. The procedure should be closely followed.

Solutions Required

(1) *Sodium Hydroxide Solution*—40% w/v in water.
(2) *Sodium Persulphate Solution*—5% w/v in water.
(3) *Acid Mixture*—Hydrochloric acid (S.G.1·18), 5 vols.; nitric acid (S.G.1·42), 3 vols.; water 2 vols.
(4) *Sulphuric Acid*—12·5% v/v sulphuric acid (S.G.1·84) in water. This solution is approximately 4·5 N.
(5) *Nitric Acid*—Nitric acid (S.G.1·42), 1 vol.; water, 1 vol.
(6) *Suppressing and Deoxygenating Solution*—Dissolve 15 g. of hydrated sodium sulphate in water, add 15 ml. of an 0·5% w/v aqueous gelatin solution and dilute to 100 ml. with water. Powdered edible gelatin was found most satisfactory.

Procedure

Place 250 mg. of sample in a 100 ml.* tall-form beaker, covered with a watch-glass. Cautiously add 5–7 ml. of acid mixture, with cooling, if necessary. When the initial reaction ceases, run in from a burette 8·5 ml. of the sulphuric acid solution. Support the watch-glass on a glass hook and evaporate until the *first definite signs of fuming* appear. Remove from the hot-plate, wash down the watch-glass and sides of the beaker with about 10 ml. of warm water and again evaporate just to *distinct fumes*. Cool, wash the watch-glass and hook and remove. The volume of the solution should then be about 15 ml. Heat until all salts are in solution, adjust the volume (by evaporation or dilution) to about 10 ml., add 3·0 ml. of the sodium persulphate solution and boil gently for 1–2 minutes after the yellow colour of oxidised chromium or a deep brown manganese precipitate appears, then digest at a moderate temperature for a further 5 minutes.

*It was found advantageous to have 25 ml. graduation marks painted or etched on all the 100 ml. beakers in use.

In the meantime, transfer 7·7 ml. of the sodium hydroxide solution into a second 100 ml.* tall-form beaker using a graduated pipette. Make a *dropwise addition* of the *hot* oxidised chromium solution to the sodium hydroxide solution, *swirling* the latter throughout the addition. Wash out the beaker which contained the acid solution, with warm water, and transfer the washings to the alkaline solution until the latter has a volume of about 25 ml. Place a funnel, together with a Green's Hyduro 81F, 7-cm. filter paper, in the original beaker. Gradually heat the sample so as to bring to the boil in about 5 minutes and then boil gently for 1–2 minutes. Filter through the Green's 81F paper into the original beaker. Wash out the beaker and then wash the precipitate four times with warm water, using a micro wash bottle with a fine jet. The filtrate, containing the chromium as sodium chromate, should have a volume of about 40 ml. Add a bumping bead, evaporate to a volume of between 18 and 20 ml., cool to 20° C. and transfer to a 25-ml. graduated flask, washing the beaker with 1·5 ml. of suppressing solution followed by a suitable volume of water. Dilute to the mark, mix well and allow to stand for about 5 minutes. Record the polarogram† over the range −0·2 to −1·2 volts using maximum damping and zero counter current. The following is an approximate indication of the useful sensitivities. (If a small amount of copper has passed into the alkaline filtrate a little counter current can be used to advantage).

0·15% chromium	1/10
0·30% chromium	1/20
0·45% chromium	1/30

Modification in the Presence of Copper as in HID RR77 Type Alloys

Place 250 mg. of sample in a 100-ml. tall-form beaker and add 5–6 ml. of 50% v/v nitric acid, followed by 8·5 ml. of 4·5 N sulphuric acid run in from a burette. Support the watch glass on a glass hook and evaporate to the first signs of fumes, cool, wash and remove the watch-glass and hook and dilute to about 30 ml. Add 2 drops of concentrated nitric acid and boil to complete solution of the salts. The volume of the solution should then be about 25 ml. Electrolyse the warm solution for 20 minutes, using a current of 2 amp. at 4–5 volts. A stationary, cylindrical platinum gauze cathode and spiral anode are suitable.‡ Stir the solution by occasional movement of the beaker. Remove the electrodes with the current still on and wash with water using a coarse micro-jet. With a watch-glass and glass hook in position, evaporate the solution until *the first definite signs of fuming* appear and continue with the method as before from solution of the sulphates and subsequent oxidation of the chromium.

Some typical average results, compared with standard volumetric figures are given in Table II.

Calibration

Process a chromium-free alloy of similar type and, if possible, of similar composition to that being analysed, and make a chromium addition of about the same order as that expected in the sample, immediately after solution of the alloy in acid mixture. A drop or two of hydrogen peroxide solution is also added to reduce the

† All the experimental work described was carried out using the standard, Cambridge instrument (Cat. No. 91311) with normal mercury pool anode.
‡ The cathodes used were about 25 mm. diameter and 18 mm. high.

continued on page 326.

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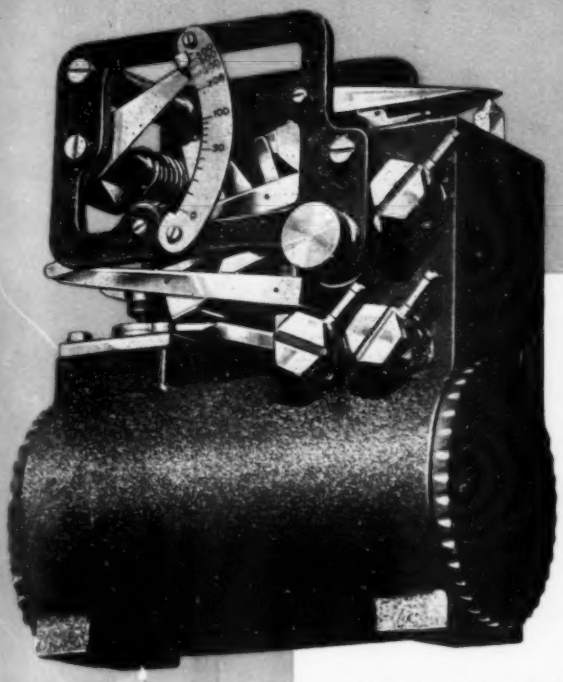

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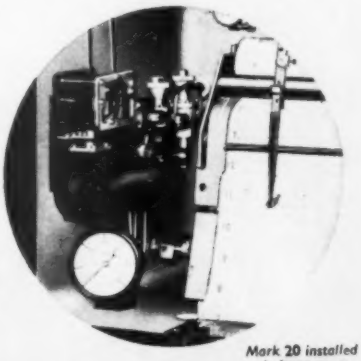

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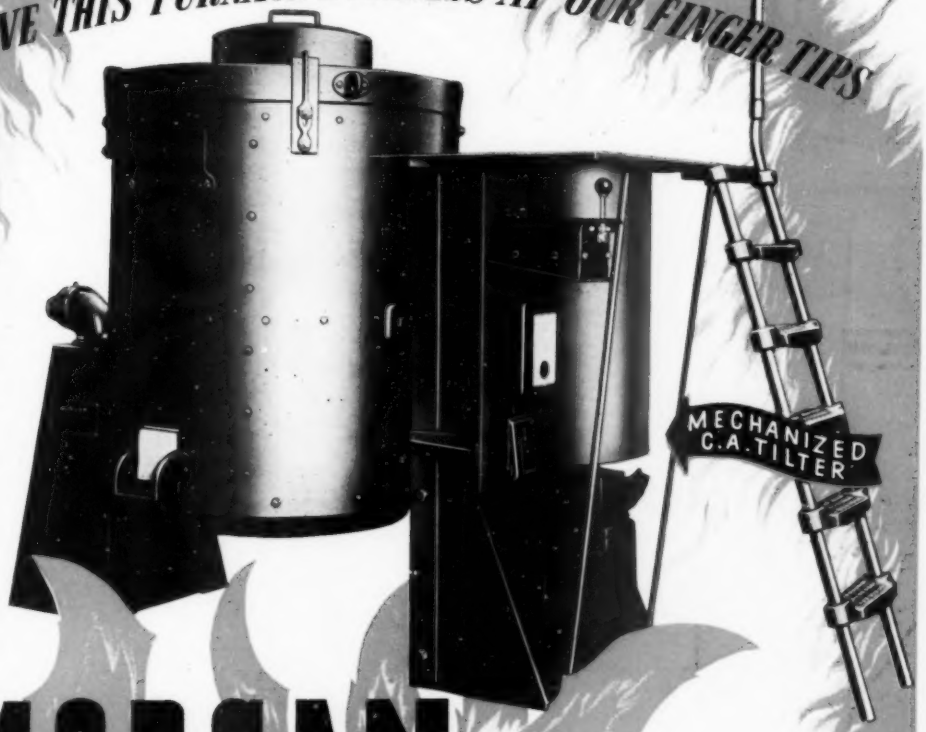

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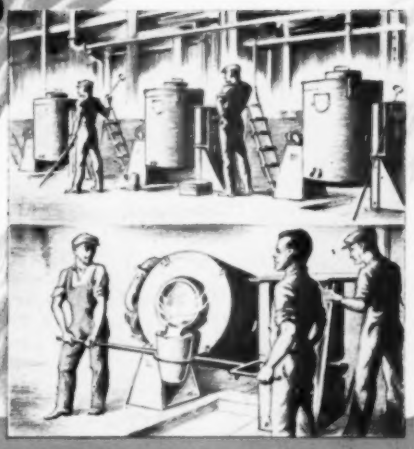

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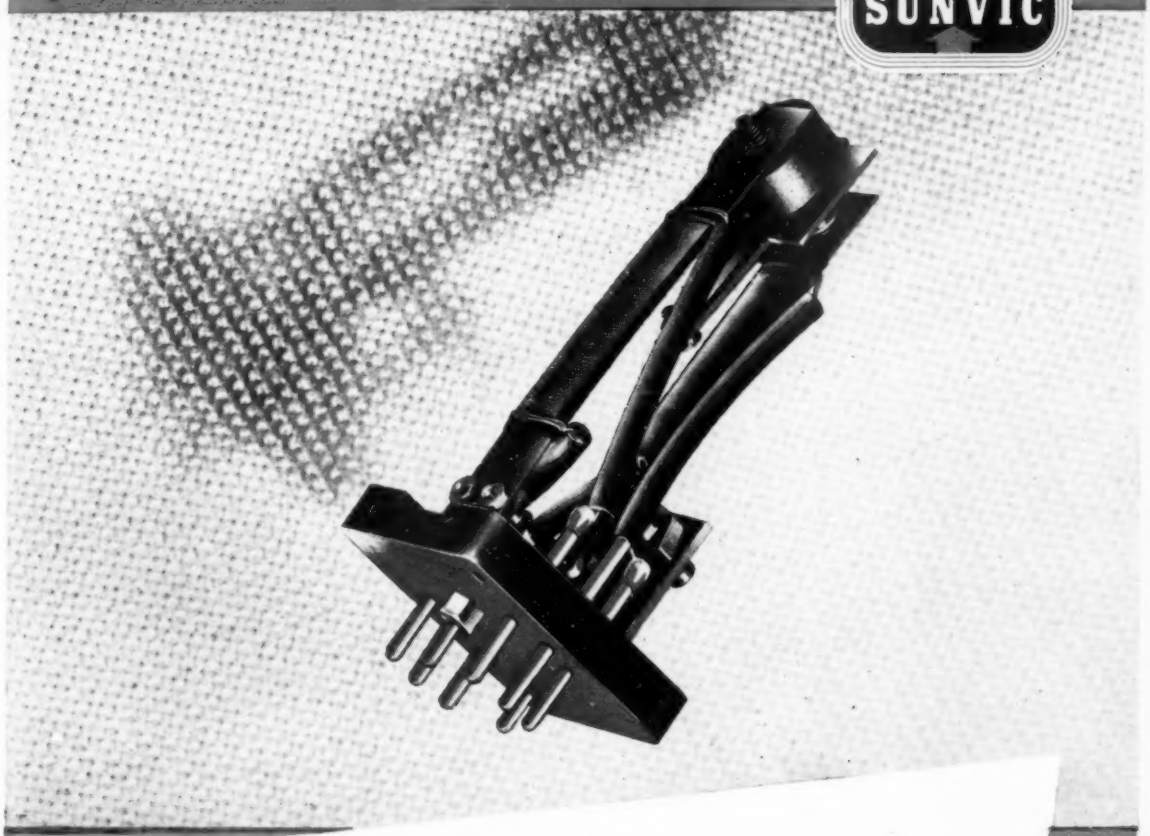

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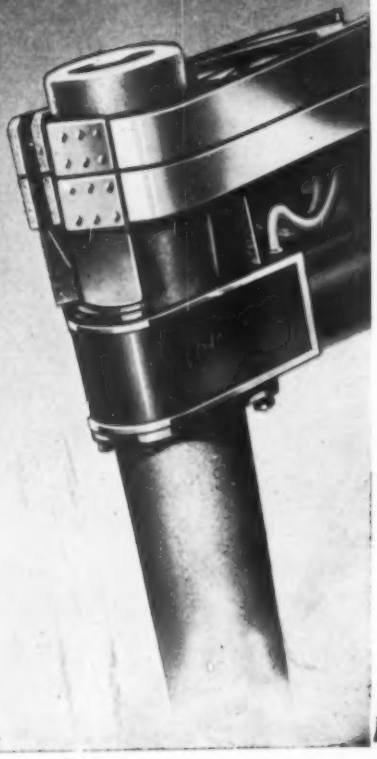

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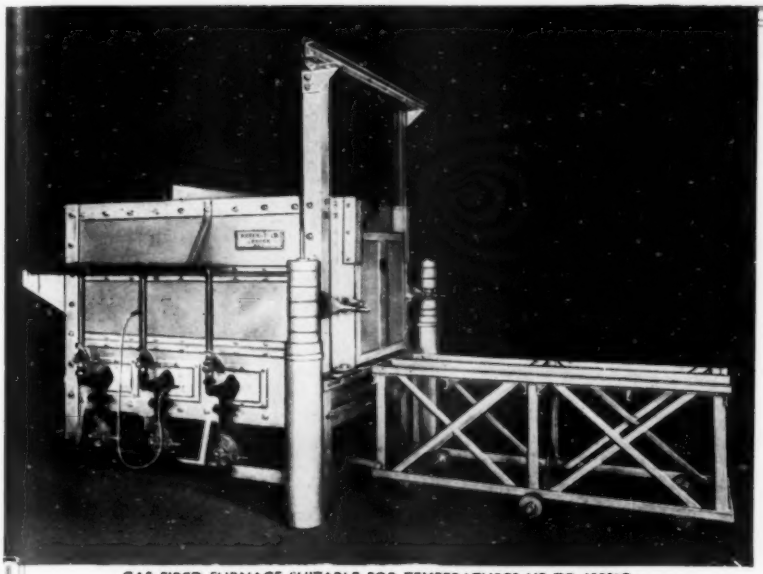

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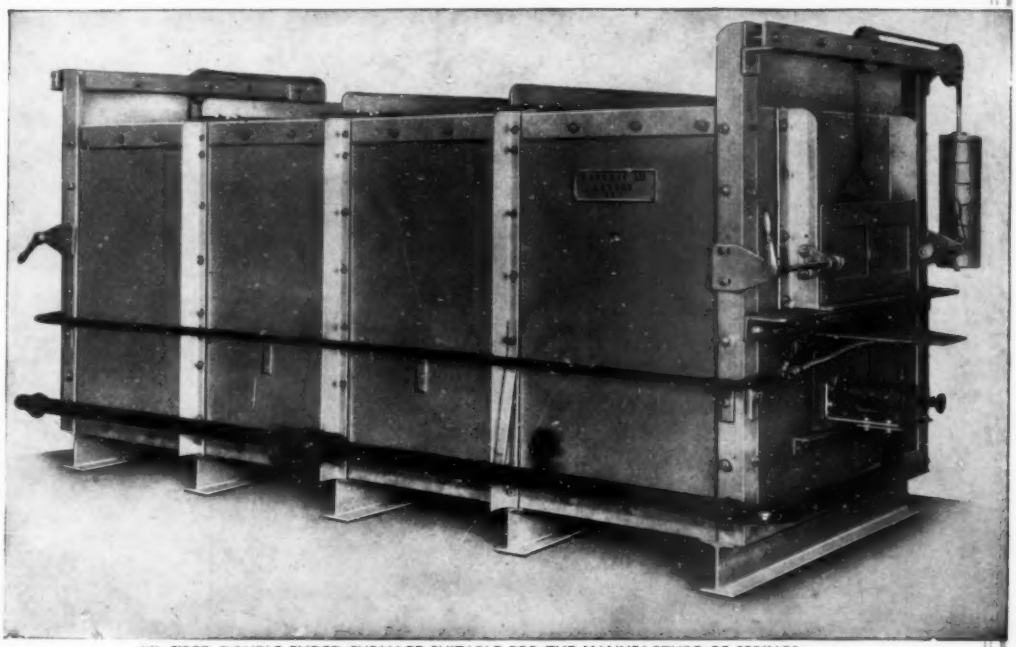

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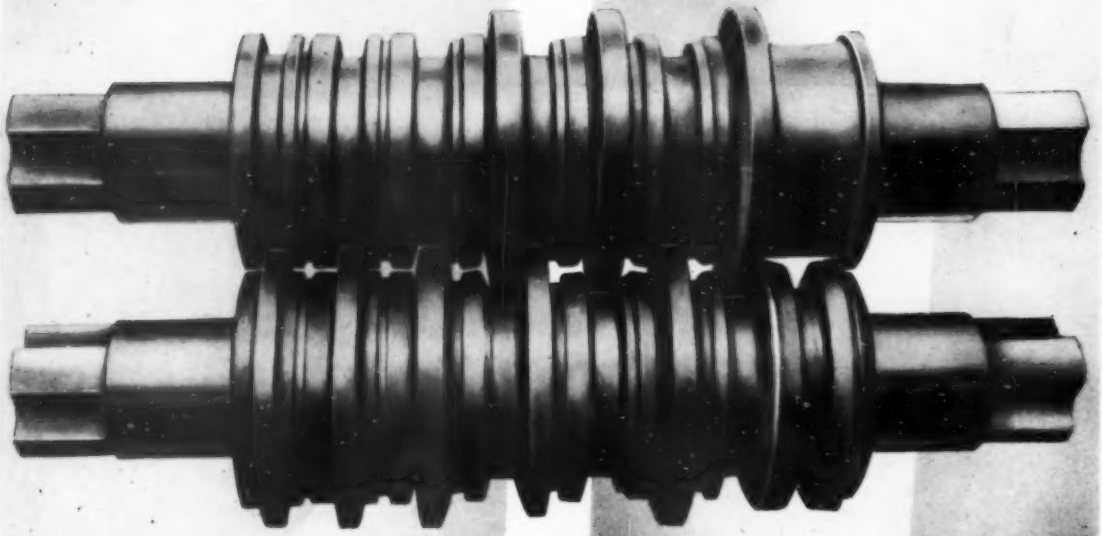

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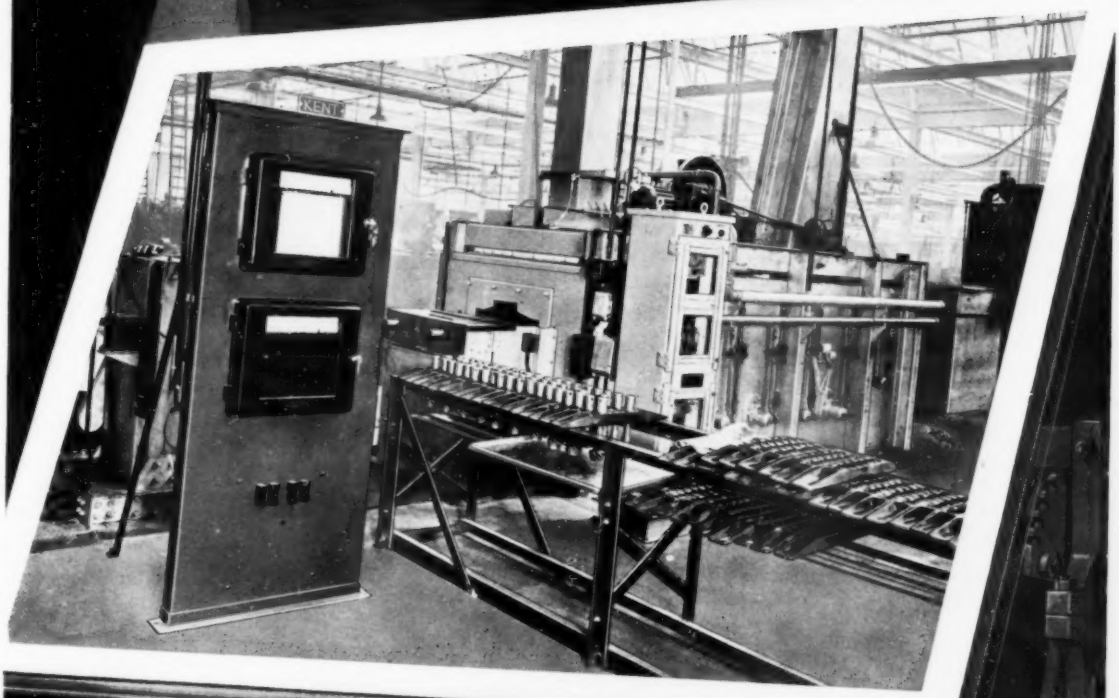

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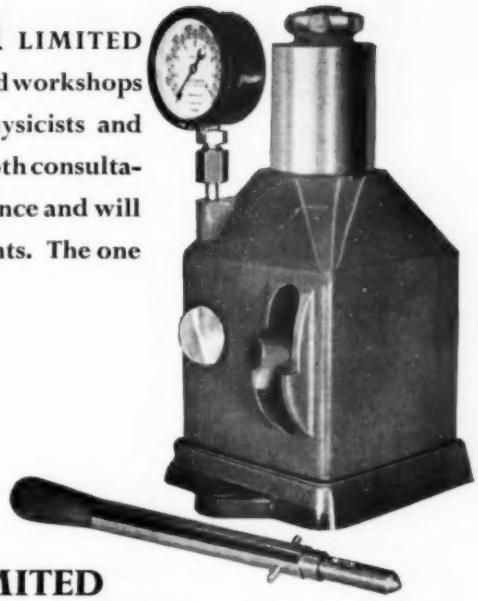

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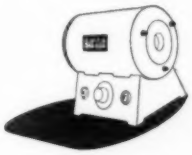

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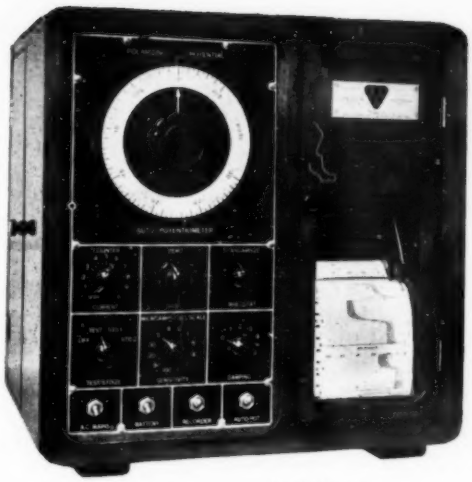

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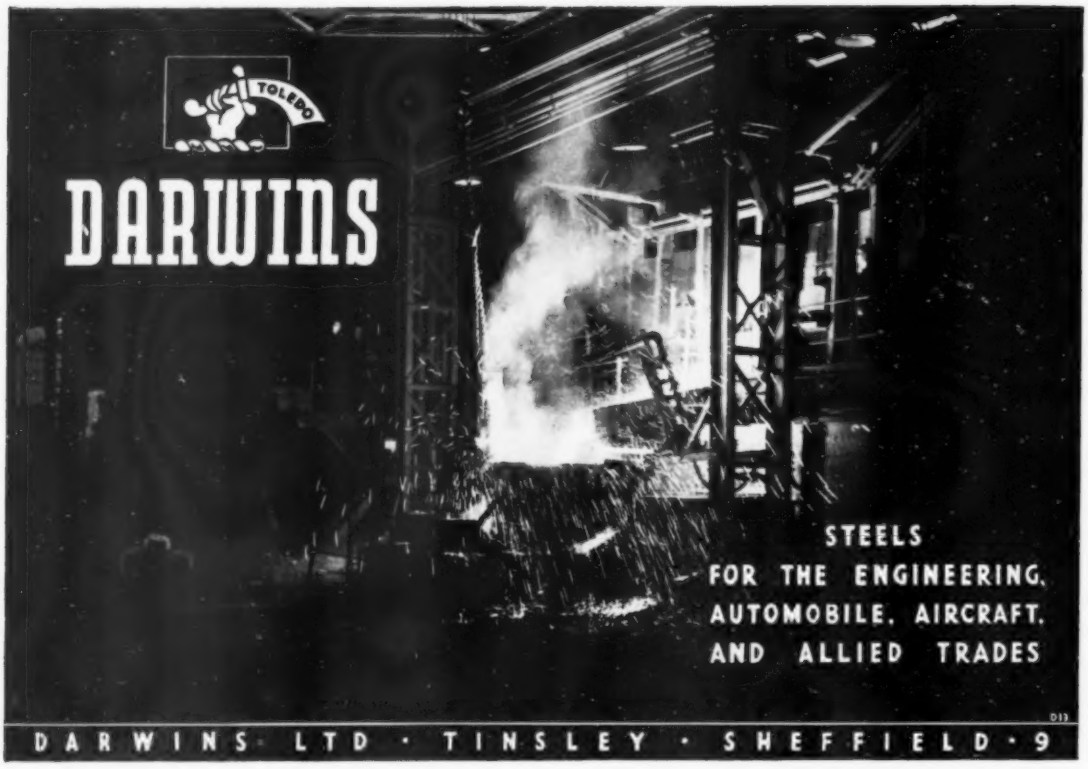

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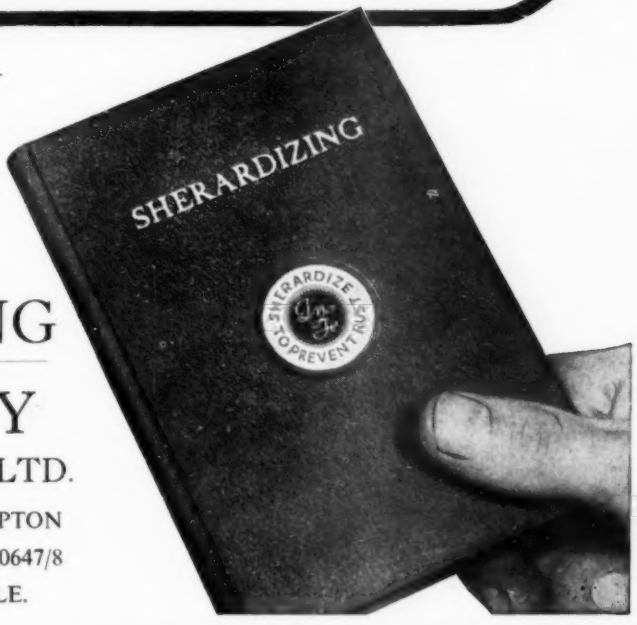

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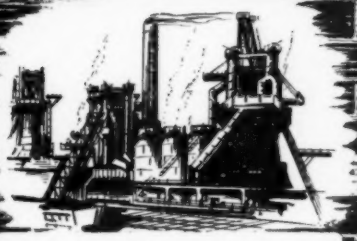

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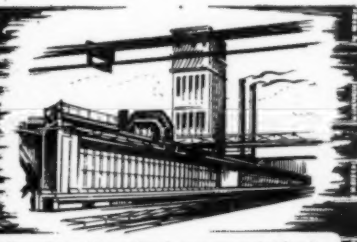

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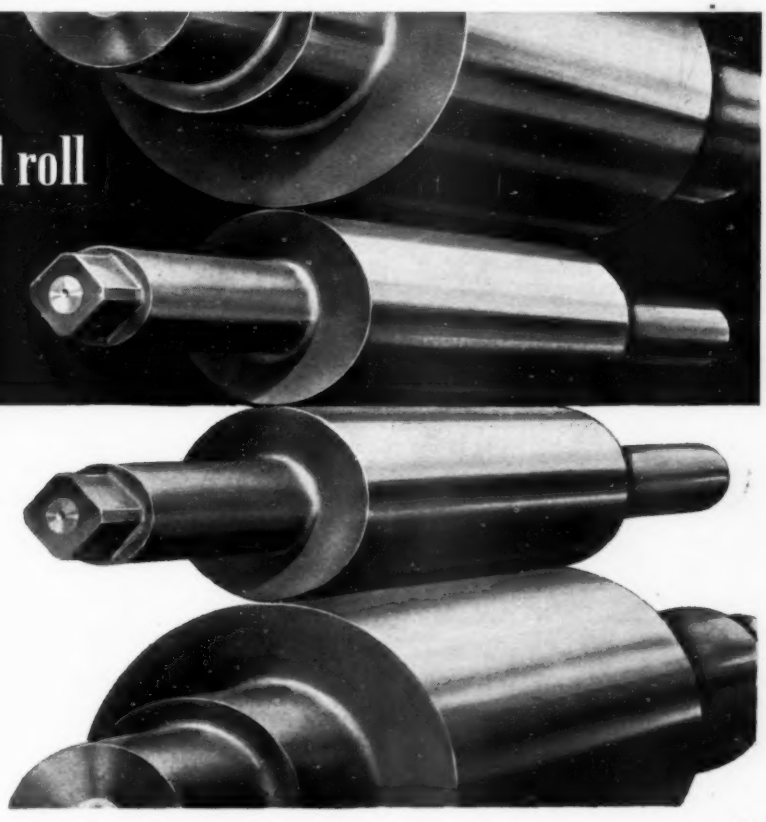

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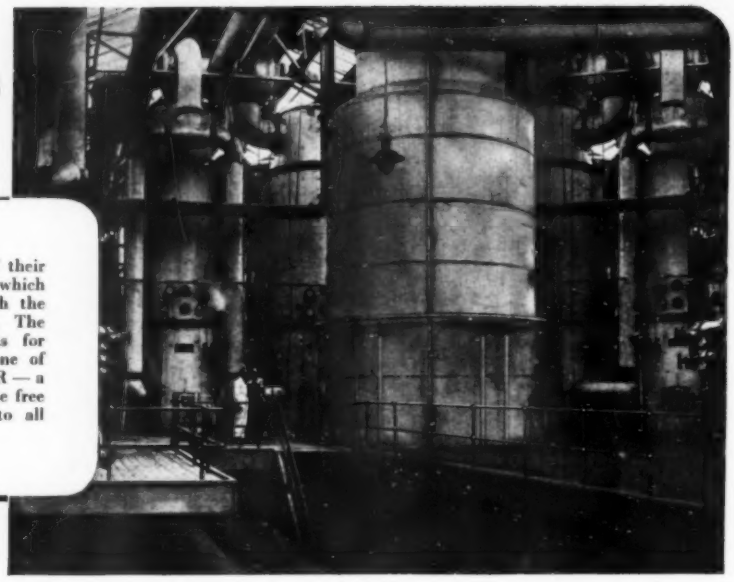

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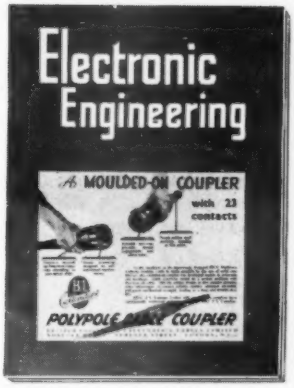

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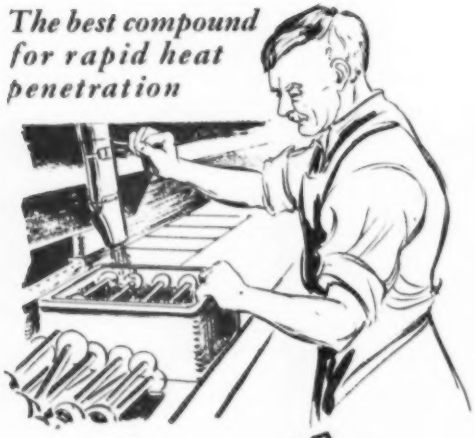

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INDEX TO ADVERTISERS

	Page
Allen, Edgar, & Co., Ltd.	29
Almin, Ltd.	42
Aluminium Union, Ltd.	—
Andrews, Thomas, & Co., Ltd.	63
Armstrong Whitworth & Co., Ltd.	48
Ashton, N. C., Ltd.	328
Association of Light Alloy Refiners	60
Baker Platinum, Ltd.	—
Beck, R. & J., Ltd.	—
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Bolton, Thomas, & Sons, Ltd.	328
Bradley & Foster, Ltd.	10
Brayshaw Furnaces & Tools, Ltd.	19
British Aluminium Co., Ltd.	—
British Acheson Electrodes, Ltd.	46
British Driver-Harris Co., Ltd.	—
British Electrical Development Association	16
British Electro Metallurgical Co., Ltd.	—
British Furnaces, Ltd.	—
British Iron & Steel Federation	61
British Oxygen Co., Ltd.	2
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Calorizing Corporation of Great Britain, Ltd.	60
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Darwins, Ltd.	54
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Doncaster, Daniel, & Sons, Ltd.	59
Dunford & Elliott (Sheffield), Ltd.	41
Durrans, James, & Sons, Ltd.	44
Electric Furnace Co., Ltd.	11
Electrothermo Meters Co., Ltd.	3
Electromagnets, Ltd.	62
Electronic Engineering	61
Electric Resistance Furnace Co., Ltd.	—
English Electric Co., Ltd.	1
English Steel Corporation, Ltd.	—
Ether, Ltd.	15
Expert Tool Heat Treatment, Ltd.	54
Felix Kramarsky Corp.	62
Fielding & Platt, Ltd.	—
Firth, Thomas, & John Brown, Ltd.	—
Flather, W. T., Ltd.	26
Fletcher, Miller, Ltd.	59
Follsain-Wycliffe Foundries, Ltd.	—
General Electric Co., Ltd. (Fluorescent Lighting)	—
General Electric Co., Ltd. (Furnaces)	6
General Metallurgical & Chemical, Ltd.	53
General Refractories, Ltd.	39
Gibbons Brothers, Ltd.	49
Girdlestone Pumps, Ltd.	—
Griffin & Tatlock, Ltd.	56
Grundy, B., & Co., Ltd.	—
G. W. B. Electric Furnaces, Ltd.	27 & 62
Hadfields, Ltd.	—
Hale & Hale (Tipton), Ltd.	Back Cover
Halesowen Steel Co., Ltd.	8
Hallamshire Steel & File Co., Ltd.	—
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High Duty Alloys, Ltd.	—
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Humphreys & Glasgow, Ltd.	21
Ilford, Ltd.	—
Imperial Chemical Industries (Cassel), Ltd.	7
Imperial Chemical Industries (Metals), Ltd.	—
Incandescent Heat Co., Ltd.	Inside Back Cover
Industrial Pyrometer Co., Ltd.	—
Integra Co., Ltd.	36
J.L.S. Engineering Co., Ltd.	22
Kasenit, Ltd.	47
Keeton, Sons, & Co., Ltd.	62
Kelvin & Hughes (Industrial), Ltd.	—
Kent, George, Ltd.	Front Cover
Kingscliffe Insulating Products, Ltd.	18
Kodak, Ltd.	—
Langley Alloys, Ltd.	—
Loewy Engineering Co., Ltd.	—
Magnesium Elektron, Ltd.	38
Makin, J. & J. (Metals), Ltd.	—
Marshall, Thomas, & Co., Ltd.	—
May & Baker, Ltd.	—
McDonald Furnaces, Ltd.	37
McKechnie Brothers, Ltd.	58
Metropolitan Leather Co., Ltd.	55
Metropolitan Vickers Electrical Co., Ltd.	—
Mond Nickel Co., Ltd.	32
Morgan Crucible Co., Ltd.	43
Nash & Thompson, Ltd.	51
Newall, A. P., & Co., Ltd.	50
New Metals & Chemicals, Ltd.	—
Newton Victor, Ltd.	56
Nitralloy, Ltd.	63
Pearson, E. J., & J., Ltd.	—
Phosphor Bronze Co., Ltd.	52
Pitman, Sir Isaac, & Son	63
Pope's Electric Lamp Co., Ltd.	28
Priest Furnaces, Ltd.	—
Priestman, T. J., Ltd.	31
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Rudge Littley, Ltd.	—
Sanderson Brothers & Newbould Ltd.	17
Shandon Scientific Co.	—
Shell-Mex & B.P., Ltd.	Inside Front Cover
Siemens-Schuckert (G.B.), Ltd.	—
Slough Metals, Ltd.	63
Smethwick Drop Forgings, Ltd.	9
Smith, John (Keighley), Ltd.	—
Stanton Instruments, Ltd.	13
Stein, J. G., & Co., Ltd.	35
Sunvic Controls, Ltd.	45
Thermal Syndicate, Ltd.	8
Thermic Equipment & Engineering Co., Ltd.	12
Thomas (Richard) & Baldwins, Ltd.	5
Tinsley (Industrial Instruments), Ltd.	53
Tungum Sales Co., Ltd.	63
United Steel Companies, Ltd.	—
Universal Aluminium Co., Ltd.	63
Vaughan, Edgar, & Co., Ltd.	—
Ward, T. W., Ltd.	23
Wellman Smith Owen Eng. Corpn., Ltd.	34
Whiffen & Sons, Ltd.	—
Wickman, Ltd.	25
Wiggin, Henry, & Co., Ltd.	—
Wild-Barfield Electric Furnaces, Ltd.	51
Wincott, G. P., Ltd.	—
Woodall-Duckham Co. (1920), Ltd.	—
Zinc Alloy Die Casters' Association	40
Zinc Alloy Rust Proofing Co., Ltd.	55

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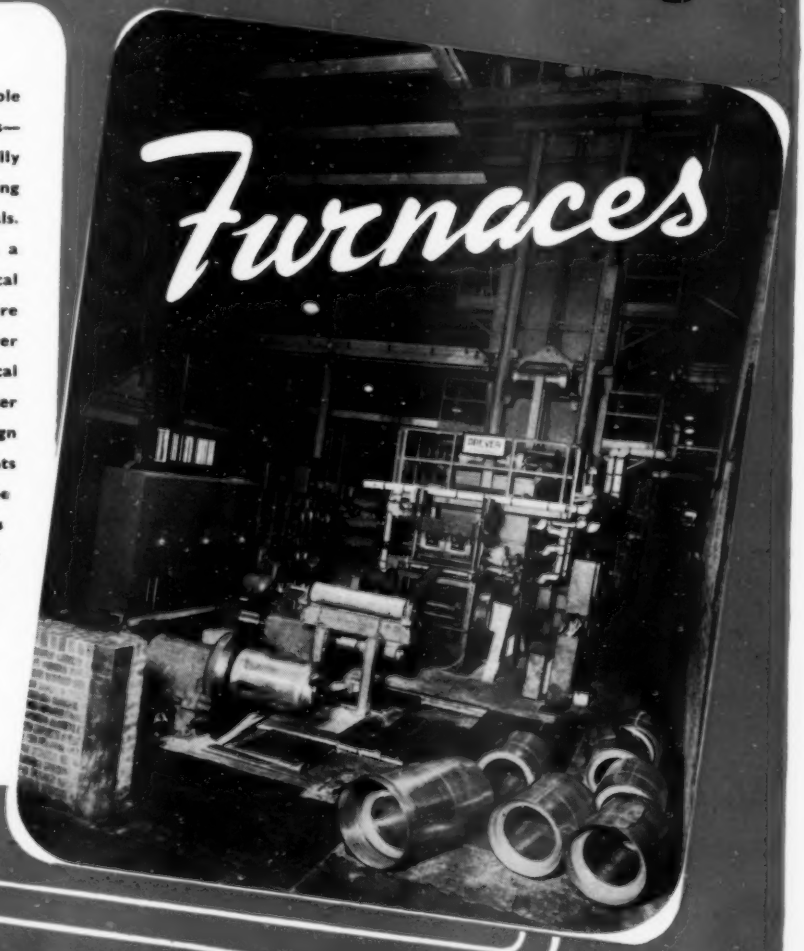

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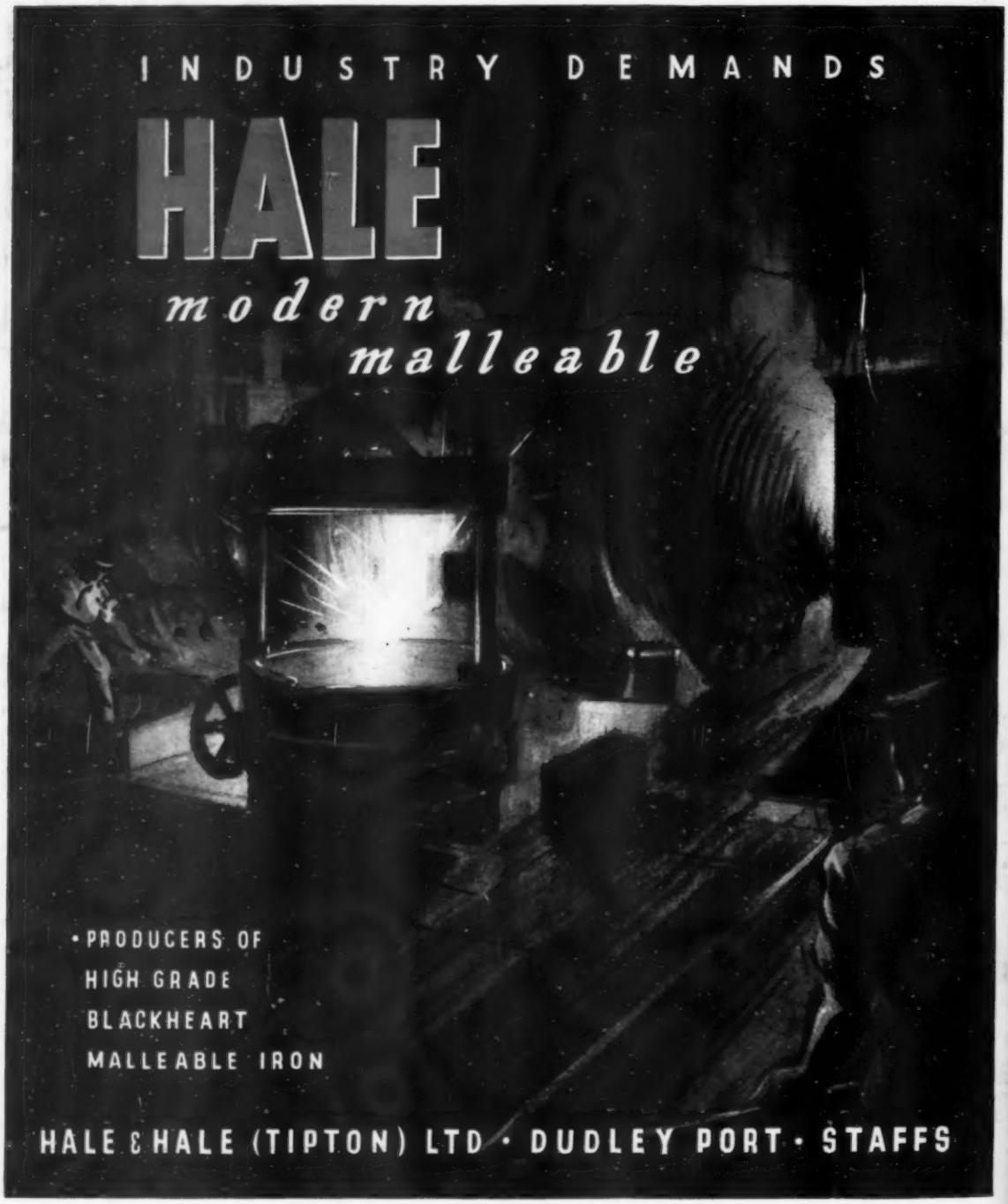

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